ON-BOARD MONITORING OF ENGINE OIL

Ryan James Clark, M.S.E Western Michigan University, 2011

Engine oil condition was quantified in a diesel engine through direct, real-time measurements of lubricant properties using an on-board oil-condition sensor. The sensor measures the lubricant temperature, density, dynamic viscosity and dielectric constant. Comparative bench-top experiments using ASTM methods or equivalent techniques validated the accuracy and precision of the lubricant property measurements from the oil-condition sensor for a specific temperature range. Bench-top experiments were also used to establish correlations between fuel contamination levels and changes in lubricant properties.

Through engine experiments, the change in the lubricant properties with respect to operating time was quantified. A correlation was found between the dielectric constant and kinematic viscosity. Specific causes for this correlation and the change in the lubricant properties with respect to engine operating time were further investigated through bench-top oil analysis.

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ON-BOARD MONITORING OF ENGINE OIL

By

Ryan James Clark

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Advisor: Claudia Fajardo, Ph.D.

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CHAPTER 1

INTRODUCTION

1.1 Engine Lubrication and Lubricant Monitoring

Internal combustion (IC) engines rely on lubricants to operate properly. Movement between IC engine components is inhibited by friction, which reduces the engine mechanical efficiency and increases component wear. A film of lubricant between engine components reduces friction and provides an additional pathway for heat transfer, thereby assisting the engine in maintaining its mechanical efficiency and preventing failure of engine components.

There are several motivational benefits from cost, environmental, and logistical perspectives to not only monitor lubricant properties, but to improve the means by which lubricant monitoring is accomplished. It is estimated that one third of the world's energy resources is consumed in applications such as the IC engine, to overcome friction [1]. The United States alone consumes approximately 2.5 billion gallons of industrial and engine oils annually [2]. Lubrication improvements, whether in lubricant monitoring techniques or in the lubricant itself can save approximately 18.6% of the total annual energy consumed by cars in the United States, which is equivalent to about 14.3 billion US dollars per year [1].

The impact that oil change intervals have from cost and environmental standpoints extends into a logistics perspective as well.

In some applications, it is required that engine oil changes be based on the oil condition instead of vehicle mileage [3]. Further yet, there are instances in which the oil condition dictates other maintenance intervals [4]. In order to determine the condition of the engine oil, it is usually the case that oil samples are sent to off-site laboratories.

Laboratory results can be either normal or abnormal, requiring either a resample or an oil change. The majority of the oil samples analyzed by these laboratories are deemed normal [3]. This shows the significant impact that on-site or on-board analyses would have in these applications. Such analysis would reduce the number of samples sent to off-site laboratories, and the involved costs. Continuous oil condition outputs from on-site analyses would also reduce shipping labor and sampling error. An additional advantage of in-situ oil analyses is that it would reduce the delay between sampling and receiving a response from the laboratory. Equipment operators would benefit from faster response times; that is, hours instead of days from the time samples are collected.

Lubricant monitoring techniques generally measure engine operating parameters; few monitor the lubricant properties. These parameters are continuously recorded during vehicle operation and used in an algorithm to estimate the condition of the oil. The problem with using engine operating parameters is that they do not monitor the physical properties of the lubricant directly; therefore critical problems such as fuel contamination can be overlooked. Excessive lubricant contamination may lead to dramatic changes in lubricant properties, preventing the lubricant from performing its required functions. Lubricant contamination may also require shorter oil change intervals than usual, which if not completed may result in catastrophic failure of engine components. Monitoring techniques that quantify changes in the physical properties of the lubricant are likely to

give a more accurate reading of the oil condition, thereby reducing oil consumption and providing the means to diagnose component failure.

As cost effective as an in-situ oil analysis system might be, there are challenges associated with its development and implementation; for example, deciding which of the lubricant properties should be monitored and knowing at which point they have reached a threshold value beyond which the lubricant is no longer able to perform its functions. These are challenging tasks because there are multiple lubricant properties that can be monitored, each of which impacts a specific lubrication mechanism.

1.2 Goals and Objectives

The goal of this research project was to quantify the quality of engine oil through direct, real-time measurements of the lubricant physical properties in an engine using an on-board oil condition sensor. The objectives of the project were to:

- 1. Validate the accuracy and precision of the lubricant properties measured by the on-board oil condition sensor. The sensor measures the oil temperature, density, dynamic viscosity, and dielectric constant. The values were validated by comparing them to results obtained through ASTM standard methods or complementary bench-top testing.
- 2. Establish correlations between contamination levels and changes in lubricant properties. Tables 1 and 2 show possible condemning limits for fuel and soot contamination, respectively. However, these values are not specific to an engine

or fuel type and were therefore used as guidelines for selecting reasonable contamination levels to test and correlate to changes in lubricant properties.

| Contaminant | Condemning Limit 1 | Condemni | ng Limit 2 | Condemning Limit 3 |
|-------------|--------------------|-------------|------------|---------------------------|
| | | Minor | 1.5-4.5% | |
| Fuel | 0.5-1.5% | Significant | 5-7.5% | 2.5% to 5% max |
| | | Excessive | >7.5% | |

Table 1: Possible fuel contamination limits by volume [5]

| Contaminant | Condemning Limit 1 Condemning Lim | | ng Limit 2 |
|-------------|-----------------------------------|-------------|------------|
| | | Minor | 1-1.9% |
| Soot | 0.2-0.9% | Significant | 2-4.5% |
| | | Excessive | >5% |

Table 2: Possible soot contamination limits by weight [5]

To establish these correlations with respect to fuel contamination, the lubricant properties of oil samples manually contaminated with various amounts of fuel were measured. Changes in the lubricant properties from a standard baseline were correlated to the amount of fuel contamination present in the sample. In addition, the changes in the lubricant properties were compared to published thresholds of these properties to assess if a specific contamination level corresponds to a published threshold.

3. Quantify how lubricant properties change with engine operating time. The temperature, kinematic viscosity and dielectric constant of the lubricant were monitored continuously during engine operation with the on-board oil condition sensor.

4. *Identify potential causes for any detected changes in oil properties*. TBN, soot, and flash point measurements were conducted on oil samples from the engine to investigate specific, potential causes for the change in the lubricant properties.

Further details of the experiments designed to accomplished objectives (1) through (4) will be presented in Chapters 2 through 4. The following sections will provide background information necessary for interpreting the findings of this research: from details on oil formulation and mechanisms for oil degradation to the relation between lubrication mechanisms, lubricant properties, and monitoring techniques.

1.3 Engine Oil Formulation and Oil Degradation Process

In order to appreciate the significance of the relation between lubricant properties and lubrication mechanisms, a basic understanding of oil formulation and degradation processes is necessary. Engine lubricants contain a base stock and additives. The base stock contributes to the performance of the lubricant package in numerous ways: thermal and oxidation stability, viscosity, volatility, and ability to dissolve additives and contaminants. The American Petroleum Institute (API) classifies lubricant base stocks into five groups. Group I-III are based on the saturate and sulfur content and the viscosity index [6]. The viscosity index (VI) describes the relationship between changes in viscosity with temperature. A high VI (e.g., 150) indicates that the viscosity exhibits a small change for a given change in temperature. Group IV are polyalphaolefins (i.e. synthetic oils). Group V includes esters and other base stocks not previously classified [6].

Each base stock group is either a mineral oil or synthetic oil. Mineral oils are manufactured from distilling and refining crude oils, which are primarily hydrocarbons with oxygen, nitrogen, or sulfur compounds. The distillation process separates the crude into compounds with similar volatility. The refining process removes undesired compounds from the crude. The molecular structure, which impacts the lubricant properties, is dependent on the source of the crude [7]. The flow properties of the base stock provide an indication of the type of molecules in the base stock. The types of molecules are primarily paraffins, naphthenes, and aromatics. Paraffins, which contribute to a high VI, have excellent temperature and oxidation stability, whereas aromatics exhibit poor performance in this area. On the other hand, aromatics are excellent solvents for additives [7].

Synthetic base stocks (e.g., polyalphaolefins) are man-made, thereby having a controlled molecular structure. Synthetic base stocks are created to have a single or few compounds with desired properties, whereas mineral oil base stocks are a mixture of compounds, some of which have undesired properties. Both synthetic base stocks and mineral oil base stocks are formulated with additives.

Additives are included in lubricant formulations to enhance the performance characteristics of the lubricant. The additives include antioxidants, anticorrosive agents, detergents, dispersants, viscosity index improvers (VII), and many others. Antioxidants and anticorrosive additives enhance the lubricant's oxidation stability. The goal of detergents and dispersants is to maintain deposits in suspension, inhibit reactions that promote oil oxidation, and neutralize acidic products from sulfur compounds [8]. VIIs improve the viscosity index of the lubricant, reducing the change in viscosity as the

temperature changes. This is significant in engine applications because engines need to operate reliably over a wide temperature range. Both the base stock and additives are carefully selected in order to minimize the rate of degradation of the lubricant in its application.

Nonetheless, the lubricant composition will eventually change due to oxidation and contamination. The process by which engine oil degrades is insightful in deciding how to monitor the lubricant and which lubricant properties to monitor. Oxidation of engine oil occurs when the oil reacts with oxygen. The initial stage of this reaction is slow and is dependent on temperature and oxygen partial pressure in addition to traces of transition metal ions which can increase the rate at which this initial stage occurs.

As the engine oil reacts with oxygen, the carbon and hydrogen bond break, yielding the formation of a radical. This radical reacts irreversibly with the oxygen to form additional radicals [9]. The radical chain reaction continues until the hydrocarbon is completely consumed or other species such as antioxidant additives interfere with the reaction, thereby terminating it. In addition to the formation of radicals, oxidation forms corrosive acids and other by-products [9]. Acids will react with other lubricant molecules, producing molecules of greater molecular weight, increasing the viscosity. The significance of oxidation is that the radicals and acids break-up the hydrocarbon chain [10]. The impact is not detrimental until a large percentage of the oil has oxidized. As the hydrocarbon chain is broken, it loses its capability to carry a load between the moving surfaces; that is, the oil loses its lubricating properties [10]. Note that oil degradation is not limited to chemical changes in the oil but also results from contamination by fuel and combustion by-products such as soot. The effects of

contamination on lubricant degradation will be expanded upon in the following section through a discussion on lubrication mechanisms.

1.4 Lubrication Mechanisms

It has been emphasized that an understanding and advancement in engine lubricant monitoring will be beneficial from cost, environmental, and logistical perspectives.

Engine oil formulation and the lubricant degradation process have been discussed, setting the foundation for an introduction into the basic understanding of lubrication mechanisms and the factors that adversely affect them, promoting or enhancing oil degradation.

The three primary lubrication mechanisms are hydrodynamic, boundary, and mixed lubrication. Hydrodynamic lubrication occurs as the lubricant flows into the loaded zone through the movement of an engine component (e.g., a rotating shaft in a bearing). The loaded zone bounds the lubricant film in a wedge shape, developing pressure in the lubricant, which keeps the engine components completely separated. The pressure in the lubricant, (i.e., hydrodynamic pressure), depends on the viscosity of the oil and the speed at which the oil is squeezed into the loaded zone. The viscosity is the only property of the oil that affects hydrodynamic lubrication. [11]

In some locations within an IC engine, under normal operating conditions, the engine components are not completely separated by an oil film. An example is the piston-cylinder interface, when the piston changes direction from top to bottom-dead-center or the valve-cam interface [12]. These conditions result in boundary friction. In such locations, the asperities of the engine components come in contact. If there is no oxide film on the parts extreme friction may develop, leading to wear and enabling components

to adhere under high load operation [11]. Boundary lubrication is highly dependent on the properties of the lubricant other than the viscosity (e.g., the additive concentration). The additives in the lubricant may be adsorbed or chemisorbed onto a metal surface. Adsorption depends on the electronic structure of the molecule. The disadvantage of this process is that adsorbed molecules may desorb if heated to a critical temperature or a more strongly adsorbed molecule is present in the lubricant. Chemisorption occurs after adsorption if there is an exchange of bonding electrons between the metal or metal oxide and the molecules in the lubricant. Molecules that are chemisorbed are more strongly adhered to the metal. Rubbing conditions have the potential to remove the adsorbed and chemisorbed films [11]. Once removed, and if the oxide film has been removed as well, there will be severe wear and potential for seizure and corrosion of the engine component. The occurrence of mixed lubrication, in which hydrodynamic and boundary lubrication occur in a given location, is prevalent in IC engines. An example is journal bearings or any engine parts in relative motion with a lubricant between them [12].

As has been mentioned, lubrication mechanisms are affected by engine operating conditions. Engine operating conditions are defined in terms of the vehicle driving cycle, engine type, engine design, and environmental conditions. From an engine lubrication perspective, severe driving cycles encompass frequent short trips, particularly during cold weather, stop-and-go driving, cold-start and high speed-high load driving. Each of these driving cycles impacts the different types of lubrication mechanisms, ultimately promoting oil degradation.

Short trips, particularly during cold weather, and stop-and-go driving, are detrimental to regimes involving hydrodynamic lubrication. Consider the area between the piston and

the cylinder wall. As the piston moves up and down it also rocks back and forth as it moves from top to bottom dead center. This phenomenon, also known as the "heal-to-toe" transition, produces an inclined surface with respect to the cylinder wall, thereby bounding the lubricant film in a wedge shape. The pressure developed between such surfaces gives the film its load carrying capacity, thereby keeping the surfaces (i.e., the piston and cylinder wall) separated to prevent wear. During stop-and-go driving, the piston is unable to maintain a constant minimum speed, which diminishes the pressure in the oil film and adversely affects the hydrodynamic lubrication mechanism.

The aforementioned driving cycles also promote the accumulation of certain contaminants. Frequent short trips, particularly during cold weather, promotes water and fuel accumulation in the crankcase because the engine operating temperature is not maintained at a high level long enough to induce evaporation. These contaminants, if excessive in amount, will affect the boundary lubrication regimes. In addition, accumulated water induces base oil oxidation, accelerating a reduction in the additive content and ultimately leading to corrosion on engine part surfaces. Water will also extract acids and additives out of the oil, leading to the formation of emulsions [13]. Emulsions restrict oil flow by blocking passages in the oil gallery, leading to wear on engine parts due to insufficient amounts of oil reaching these components. In regards to oil contamination by fuel, the critical oil film thickness is reduced, resulting in cylinder, ring, and piston wear.

Another severe driving condition is high load operation because it increases the engine operating temperature. High-temperature operation promotes oxidation, causing premature degradation of the oil base-stock. The compounds generated due to oil

oxidation may deposit on the piston rings and grooves, causing ring-sticking and promoting blow-by. Excessive blow-by will in turn decrease the brake (output) engine power and increase the specific fuel consumption. By-products from oxidation will eventually lead to depletion of the additive package, negatively affecting boundary lubrication. The experimental data gathered in this research was measured in a diesel engine under high-load operation. These data will be discussed in Chapter 4.

The second operational parameter that impacts the lubricant mechanisms is engine type and design. Diesel engines produce more soot than gasoline engines. Soot is primarily formed from the carbon in diesel fuel [12]. The molecular structure of soot causes it to have an affinity for other species, so it either collects on engine components or eventually agglomerates, forming larger soot particles and eventually increasing the oil viscosity. This makes soot a detrimental contaminant. The production of soot is a complex process, dependent on combustion characteristics and fuel composition. An example in which engine design affects lubricant mechanisms is the incorporation of exhaust gas recirculation (EGR) systems. As the name suggests, a percentage of the exhaust gas is re-circulated back into the engine intake manifold. This reduces the engine operating temperature, which in turn reduces oxide or nitrogen (NOx) emissions. The disadvantage, from a lubrication perspective, is that the lower operating temperature actually promotes development of particulate matter leading to increased soot contamination.

The final parameter which impacts the lubricant mechanisms is the environmental conditions the engine might be subjected to during operation. Harsh environmental conditions accelerate lubricant degradation and contamination. For example, operating

vehicles in dusty environments increases the likelihood of introducing abrasive particles (e.g., silicon) that contribute to the wear of power train components.

This introduction to the complexity of lubrication mechanisms and the numerous operational parameters that simultaneously affect these mechanisms provide a foundation for understanding the significance of lubricant monitoring. The following section will present background information on lubricant properties, including their expected trends during oil degradation.

1.5 Engine Oil Properties

a. Viscosity

Viscosity is a critical lubricant property, and the most important property in hydrodynamic lubrication. In generic terms, it has been defined as a measurement of the resistance to flow. There are two types of viscosity: dynamic and kinematic. Dynamic viscosity is the fluid property that relates shearing stress to fluid motion. It is typically measured in units of centipoise (1cP = 1mPa·s). Kinematic viscosity is the ratio of the dynamic viscosity and the fluid density. It is typically measured in units of centistokes (1cSt = 1 mm²/s). This ratio is only applicable to Newtonian fluids. Such fluids maintain the same viscosity regardless of shear rates. Non-Newtonian fluids exhibit a decreased viscosity as the shear rate increases. Most engine lubricants contain viscosity index improvers, making the lubricant non-Newtonian. Viscosity index improvers are additives that expand in high temperature environments and contract in low temperature environments. This behavior enables the oil to exhibit desirable viscosity over a wide temperature range. The disadvantage

of viscosity index improvers is that the molecules can be separated under high shear forces. Under such circumstances, the lubricant may not retain the required viscosity at a given temperature for proper lubrication of engine components. Heavily oxidized oils are likely to exhibit non-Newtonian behavior, whereas contaminated lubricants are likely to behave as Newtonian fluids [14].

Since viscosity is a temperature-dependent property, engine lubricants were originally manufactured as a single grade. A certain grade would be used in the winter (e.g., 15W) and a different grade would be used in the summer (e.g., 40). New winter-grade oils must exhibit a dynamic viscosity, as specified by a classification system established by the Society of Automotive Engineers (SAE) known as SAE J300, that allow minimum resistance to cranking and pumping at low temperatures. The 'W' applied to the grade implies that the oil is suitable for winter use. Summer grade oils must have a kinematic viscosity at 100°C that is within a specific range of values as specified by SAE J300. As technology improved, lubricants of different viscosities could be mixed through the addition of polymers, resulting in a multiviscosity lubricant (e.g., 15W-40). Such oil was used in this research. Multi-grade engine lubricants are formulated such that they lubricate across a wide range of temperatures.

Viscosity is affected by oxidation, soot loading and contamination by fuel, water and coolant (glycol). Viscosity is commonly measured from a method developed by the American Society of Testing and Materials (ASTM), Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids and Calculation of Dynamic

Viscosity (ASTM D 445) [15]. This method was used throughout the research to accomplish the project objectives.

b. Total Acid Number (TAN)

TAN quantifies the acid concentration in engine oil in units of milligrams of potassium hydroxide (KOH) needed to neutralize one gram of oil sample (i.e., mg KOH/g of sample). TAN measurements of oil samples taken over a period of engine operation show an increasing trend due to accumulation of acids from combustion, oxidation or contamination. Particle analysis often supplements TAN measurements in order to indicate the degradation severity or the condition of certain engine components.

A high TAN does not necessarily imply that the oil is corrosive. The potential for the oil to cause corrosion is dependent on the amount of weak and strong acids in the oil. Strong acids (e.g., sulfuric acid) are by-products of contamination and are corrosive. Weak acids (e.g., carboxylic acids) are by-products of oxidation and do not have the potential to be corrosive but can increase the viscosity of the oil [10]. For this reason, a trend in TAN values is typically considered, rather than a single value at an instant in time.

The two common ASTM methods to measure TAN are ASTM D 664 and ASTM D 974 [15]. These methods involve potentiometric and colorimetric titrations, respectively. For both tests, a titration solvent is prepared and added to the oil. The titration solvent is a mixture of toluene, isopropyl alcohol, and water. Toluene is a solvent for the oil and its constituents. The acids are solvated in the toluene, held in a mixture miscible with both toluene and water (i.e., the isopropyl alcohol) and then

dissociate. The acids are neutralized by adding a basic titrant, KOH. The strong acids readily dissociate as the KOH is added. The first titration end-point represents complete neutralization of the strong acids. The second end-point represents the neutralization of the weak acids [10]. While useful, the total acid number is not a reliable indicator of additive depletion [16]. This remark will be further explained in the following section in which the total base number (TBN) test is introduced.

c. Total Base Number (TBN)

The alternative to measuring TAN is measuring TBN, the concentration of basic constituents in the oil, since the additives in the lubricant are primarily basic constituents. The two common ASTM methods for measuring the TBN are ASTM D 2896 and ASTM D 4739 [17]. Both methods involve potentiometric titrations.

ASTM D 2896 uses a stronger acid and more polar solvent than ASTM D 4739. This is to ensure that all the bases, both strong and weak, in the additive package are titrated. For this reason ASTM D 2896 is a more appropriate test for new oils. The problem in applying this test to used oils is that used lubricants often contain weak bases (e.g., wear metals) which are products of lubricant degradation. If these weak bases react with the titrant, then ASTM D 2896 will report a falsely exaggerated base number for the used oil. ASTM D 4739 is most useful for measuring the TBN of used lubricants since it titrates with a weaker acid. ASTM D 4739 was used in this research since the majority of tests were conducted on used oil samples.

The base number will trend downward versus operating time, just as the acid number trends upward. It is valid practice to observe the point at which the acid and base numbers cross. Figure 1 illustrates such an example.

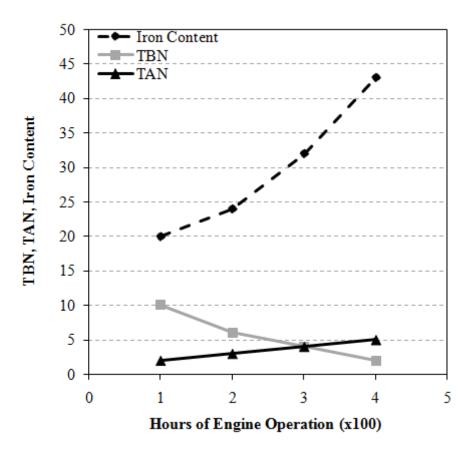


Figure 1: TBN, TAN, and iron content trend in a used lubricant

Some engine manufacturers recommend an oil change at the operating time in which the base and acid numbers intersect. As shown in Figure 1, this recommendation would be supported by the rapid increase in the iron content following the point of the acid and base intersection. Due to cost constraints and limitations on laboratory equipment availability, it may not be possible to conduct acid and base number tests along with elemental analysis. In such cases, engine manufactures or oil companies may establish rules specifying when the oil should be changed. For example, some may recommend the oil to be changed when the base number reaches a low limit, such as 3.0.

Engine lubricants with a TBN higher than other lubricants are not necessarily better performing engine oils. The critical information is the test method and the rate at which the TBN decreases. Some oils start with a high TBN, then drop and lose their neutralizing ability quickly. Other oils can start with a comparatively lower TBN that decreases at a slower rate, and maintain their alkalinity for a much longer period of time [18]. This is all dependent on the type of additives in the oil.

Studies have been conducted to model oil alkalinity in diesel engines. Researchers have shown TBN depletion to follow first-order reaction kinetics; that is, the rate at which the alkaline reserves deplete is dependent on the concentration of the reactants. Dyson and others showed that TBN varies exponentially with time according to the following equation [19]:

$$TBN_{t} = TBN_{0} - \frac{A}{r} \{1 - e^{-\left(\frac{r}{L0}\right)t}\}$$
 [Eq. 1.0]

Where:

r = rate of oil consumption in liters per hour or liters per kilometer

 L_0 = is the oil sump capacity in liters

A = TBN depletion rate constant

t = running time in hours or kilometers

Subscripts 0 and t represent initial and instantaneous values.

This model is based on first order reaction kinetics and assumes oil consumption and oil make-up to be continuous processes. The TBN depletion rate constant, A, takes into consideration engine type, operating conditions, fuel type, and oil type. A is proportional to fuel sulphur content, expressed in terms of the fuel sulphur that

reacts with the basic constituents in the oil, fuel consumption rate, and percentage of sulphur content in the diesel fuel. In [19], the goal was to model the TBN depletion rate constant for specific system variables. This study recognized mathematical models to be difficult to develop for such a variable as the TBN depletion rate constant due to the complexity of the physiochemical changes in the oil. For this reason, correlation-regression analysis was deemed as the most realistic method to model the TBN depletion rate constant in terms of system variables. The following system variables were included in this experimental study: bmep; piston speed; metal content index of oil; fuel sulfur level; oil and fuel consumption. The model was validated with a chi-square test at 99% confidence level and correlation coefficient of 0.9888 was obtained. The model was compared to experimental TBN values obtained from ASTM D 2896. The set of TBN values from the model and ASTM experiments varied by \pm 15%. This is the reproducibility limit of the ASTM method, thus making the model acceptable [19]. This case study shows that the TBN is affected by both operational parameters and the physical properties of both the lubricant and fuel. Quantifying changes in the TBN over an operational period can be suggestive to numerous reasons for the changes, leading to considerations as to how to lower the rate at which the TBN depletes. For example, using a fuel with lower sulfur content or developing more conservative driving habits.

d. Dielectric Constant

The dielectric constant is a dimensionless parameter that quantifies the magnitude of the interaction of an oscillating electric field with a molecule. It indicates the capacity of the medium (i.e., the lubricant in this case) to store a charge. The

dielectric constant of engine lubricants ranges from 2.1 to 2.8 for the base stock and from 6 to 10 for the additive package. In addition to the additive package, the viscosity and paraffinic/naphthenic content of the oil have a substantial impact on its dielectric constant. These parameters influence which material property (i.e., the polarizability or dipole moment) has the greater contribution. The electrons in naphthenic molecules are more polarizable than the electrons in paraffinic molecules [20]. That is, the electrons in naphthenic molecules exhibit a greater interaction with an electric field of a low magnitude than those of paraffinic molecules. Therefore, engine oils with a higher naphthenic content should have a larger dielectric constant.

As for the dipole moment, the base stock of the oil does not contain dipole moments, but additives and contaminants do. Molecules with a dipole moment have two physically separated charge centers. When an oscillating electric field is applied to a molecule with a dipole moment, the charge centers begin rotating in order to align themselves to oppose the field. If the electric field frequency is too high, the molecule does not have time to rotate and thus the dipole moment no longer contributes to the dielectric constant [21]. Therefore, the additives increase the dielectric constant, provided that the frequency is not too high.

Dielectric constants measured from an applied electric field at frequencies below 106 Hz show no frequency dependence in this region [21]. The applied frequency can be significant, if above 106 Hz, in comparing the results from different measuring devices. The oil condition sensor used in this research operates under varying frequencies, the smallest frequency being 25,500 Hz. This was taken into

consideration when analyzing dielectric constant results from the oil condition sensor and the bench-top instrument, which operated in a frequency range of 10,000 Hz [21].

The dielectric constant is expressed in greater detail in the Debye equation [20].

$$\frac{\varepsilon - 1}{\varepsilon + 2} = \frac{\alpha + \mu^2 / 3kT}{L\rho / 3MW}$$
 [Eq. 1.1]

Where:

 ε = dielectric constant of oil

 α = polarizability of oil

 μ = dipole moment of oil

 $k = Boltzman constant of oil (1.31 \cdot 10^{-23} Joules/degree Kelvin)$

T = temperature in degrees Kelvin

L = Avogadro's number

 ρ = density of oil

MW = molecular weight of oil

The Debye equation cannot yield an exact solution to the dielectric constant of liquids, since thermal motion is continuously changing the orientation of the dipoles [20]. Nonetheless, it is insightful in understanding a likely trend in the dielectric constant of lubricants during engine operation. During typical engine operating conditions, the oil temperature increases to approximately 100°C. The dielectric constant for hydrocarbon oils will decrease by approximately 0.0013 or 0.05 percent per degree Celsius [21]. The Debye equation indicates that the dipole moment is inversely related to temperature. This implies that as the temperature increases, the

contribution of the dipole moments, which are present in the additives, would decrease, lowering the dielectric constant. If the additives are near depletion, the state of the base oil, which does not contain dipole moments, would contribute to a decrease in dielectric constant as the temperature increases. This trend is a result of the inverse relationship between temperature and density. As the density decreases there are fewer molecules per unit area, and thus less interaction between the applied electric field and the molecules.

It has been suggested that measuring the temperature dependence of the dielectric constant will lead to useful, real-time oil quality analysis in operating systems [20]. This is because, as previously mentioned, the dielectric constant of engine oils has a dependence on the additive package and contamination (particularly due to oxidation by-products and soot contamination) in addition to temperature. Both the additives and sources of contamination (i.e., oxidation or soot) depend on temperature as well. An assessment of the temperature dependence of the dielectric constant could lead to inference on the degree of oxidation and soot contamination. Before any inference can be applied in the field, controlled laboratory experiments that measure the dielectric constant, temperature, oxidation, and soot monitoring would be required.

The error in strictly relying on the Debye equation for predicting a trend in the dielectric constant of hydrocarbons is that it does not account for the accumulation of degradation by-products or contamination. As the oil degrades, acids will make-up a larger percentage of the molecules in the oil. Consequently, there will be more interaction between the applied electric field and the lubricant, resulting in an increase in the dielectric constant. In terms of oil contamination by fuel, there will be

a small change, if any, in the dielectric constant because fuel and oil have similar dielectric constants [20]. The lubricant conductivity increases in response to soot contamination, thereby increasing the lubricant dielectric constant [23]. As the degradation process continues, heavier molecules accumulate in the lubricant, increasing the bulk density of the engine oil. As mentioned, this will increase the dielectric constant as well. These effects are summarized in Table 3.

| Parameters that increase the dielectric constant | Parameters that decrease the dielectric constant | Parameters with marginal effect on dielectric constant |
|--|--|--|
| Dipole moments (found in additives) | Increasing temperature | Fuel contamination |
| Acids from oil degradation Soot contamination | Decreasing density | |

Table 3: Parameters that influence dielectric constant behavior

It is difficult to use solely the dielectric constant to assess the condition of the oil because the condemning limits are not well established. Changes of 0.2 and 0.01 [21] have both been considered as condemning limits for the dielectric constant of engine oils. Therefore, dielectric constant measurements are likely to be most useful when supplemented with other property measurements to state the condition of the oil.

In this research, the dielectric constant was measured using a commercial instrument (i.e., BI-870, Brookhaven Instruments Corporation). These results were compared to the output from the oil condition sensor. Prior to purchasing this instrument, oil samples were sent to the manufacturer's laboratory to confirm its suitability to measure the dielectric constant of engine oils. Results will be presented in Chapter 2 Section 5d.

e. Elemental Analysis

Elemental analysis techniques, which rely on various spectroscopic methods, are useful in detecting the presence of wear metals. This approach has the potential to indicate corrosion or wear of a specific engine component based on the type of wear metals that are present. Elemental analysis was not pursued in this work due to cost limitations.

1.6 Significant Engine Oil Properties and their Limits

Disagreements exist in regards to which of the multiple parameters (e.g., viscosity, TBN, etc.) are most critical to monitor and which thresholds determine the oil change interval. Previous research from other laboratories investigated which of the physical properties of engine oil are most critical for oil condition analysis and which of the current oil monitoring devices would be most beneficial from different standpoints. In [22], manufactures of engines, transmissions, and generators were contacted for their oil change criteria, key oil quality parameters used for oil monitoring, and the rationale for identifying critical lubricant properties. This information revealed that the oil change interval was engine manufacturer-dependent. One engine manufacturer suggested elemental analysis for detecting oil contamination whereas another recommended viscosity and flash point measurements in addition to elemental analysis for determining the condition of the oil. A third engine manufacturer relied on viscosity and TBN measurements for determining the health of the lubricant.

Oil viscosity, however, was agreed upon as the most critical parameter related to both oil degradation and engine condition. TBN was considered to be a significant parameter

for determining the extent of oil degradation. Table 4 was presented at the end of the investigation to assess the cost and risk in quantifying a number of oil properties.

| Lubricant Properties | | | | |
|---|-----------------------------|----------------------------|--|--|
| Level 1: Minimum | Level 2: Intermediate Cost, | Level 3: High Cost, Lowest | | |
| Cost, Highest Risk | and Risk | Risk | | |
| Viscosity | Viscosity | Viscosity | | |
| TAN | TAN | TAN | | |
| | TBN | TBN | | |
| | | Contaminant concentration* | | |
| *Using techniques such as FTIR and wear particle analysis (WPA) | | | | |

Table 4: Level of significance for lubricant properties. Adapted from [22]

A level 1 oil analysis would involve choosing the least number of lubricant properties to measure. This analysis would be the least expensive, but would also involve the greatest risk of incorrectly assessing the condition of the oil and protection of engine components. A level 3 oil analysis would be the most expensive, but it would involve the lowest risk in possibly overlooking a problem with the lubricant.

Whereas the dielectric constant is not specifically listed as a measurable parameter to quantify oil health in the aforementioned study, additional sources list it as an alternative to conducting an elemental analysis. While elemental analysis can provide an accurate measure of wear particles and soot contamination, this technique is limited to laboratory settings. In contrast, the dielectric constant can be measured on an in-situ basis. Further yet, dielectric constant measurements, to some extent, quantify the effect of contaminants present in the oil. Based on this information and considering the objective of employing an on-board sensor to diagnose engine oil condition, measurements of viscosity, temperature, and dielectric constant were selected. The on-board sensor was then chosen

based on these considerations. The proposed condemning limits for viscosity and dielectric constant are shown in Table 5. Note that these limits are not referenced to a specific engine or lubricant type.

| Property | Condemning Limits |
|---------------------|------------------------------------|
| Viscosity @ 100°C | 10-18 cSt [5] |
| | From +25% to -18% [5] |
| | ± viscosity grade from new oil [5] |
| TBN | 0.5-4 mg KOH/g [5] |
| | -50% [5] |
| Dielectric Constant | 0.01 [21] |
| | 0.2 |

Table 5: Condemning limits for some physical properties of engine oil

In addition, TBN and flash point measurements were conducted to complement sensor output and help identify potential sources of oil degradation. Condemning TBN limits are also presented in Table 5. Although insightful, particle or wear metal analysis was not conducted due to cost constraints.

1.7 Published Results for Property Trends

Several techniques for monitoring the quality of engine oil have been developed.

Some of the case studies on these techniques reveal information about lubricant properties and their trends. The first case study reviewed discusses observed trends in oil properties as measured from commercially available and prototype sensors [23]. The research focused on evaluating the capability of the sensors to monitor the condition of in-service diesel engine oils. If the sensors proved capable, they would eliminate the

need for oil samples from a fleet of vehicles to be sent to off-site laboratories for analysis, due to their capability to provide real-time oil condition monitoring.

In [23], the authors present results obtained with a number of sensors that monitored the viscosity, dielectric constant and conductivity of engine oils. The sensors were evaluated in a laboratory setting and then on an engine test stand, using a 6.5 liter, V-8, naturally aspirated diesel engine. The engine operated eight hours a day and cooled once to 70°C to record sensor response. Oil samples were taken approximately every fifteen hours for laboratory analysis and results were compared to the output from the sensors. The following monitoring techniques, shown in Table 6, were utilized for laboratory analysis of used engine oil.

| Monitoring Techniques | | |
|--|--|--|
| Fourier Transform Infrared (FTIR) Spectrometry | | |
| Inductively Coupled Plasma-Atomic Emission Spectrometry | | |
| ASTM D 445 Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids | | |
| at 40°C | | |
| Infracal Soot Meter | | |
| Karl Fischer Titration | | |
| ASTM D 664 Standard Test Method for Acid Number of Petroleum Products by Potentiometric | | |
| Titration | | |
| ASTM D 4739 Standard Test Method for Base Number Determination by Potentiometric Titration | | |
| Gas Chromatography | | |

Table 6: Monitoring techniques for the engine oil analysis presented in [23]

These laboratory techniques provided complementary data so that the sensors could be evaluated on their capability to accurately and precisely monitor soot contamination, fuel contamination, water contamination, and oxidation by-products.

Engine experiments were adjusted to maximize soot contamination while also minimizing the effects of other sources of contamination (e.g., oxidation). To do this, the

engine oil temperature was kept below 80°C by using an external oil cooler. Minimal oxidation effects were verified through FTIR measurements of the oil samples. The percent of soot in the samples, as measured based on the conductivity sensor, showed an average difference of -0.2% in comparison with the Infracal Soot Meter [23]. Both the sensor and bench-top instrument were able to track soot concentration over time. The viscosity and dielectric sensor measured an increase in the measured properties as the soot concentration increased.

To test the sensors' ability to respond to oxidation, the engine oil temperature was raised to 150°C during operation. The dielectric constant, as measured by the sensor, increased nearly at the same rate as the oxidation by-products, as measured by FTIR, during engine operation. The viscosity sensor showed an increase in viscosity later in the testing, most likely after the antioxidant levels decreased.

To simulate fuel dilution, additional fuel was injected into the engine at a constant rate of 0.5mL per minute. The dielectric constant and conductivity showed a small change attributed to a 1.5% increase in soot concentration from the baseline, not fuel dilution. The viscosity sensor showed a leveling off of the viscosity, caused by a simultaneous increase in soot contamination and fuel contamination.

Other studies have been conducted on sensors that measure the electrical properties of engine oil. An engine oil sensor developed at Delphi Automotive Systems measures the conductivity of the engine oil [24]. The oil condition sensor was installed inside an oil drain plug, in a variety of vehicles that were driven on the highway and in the city. Oil samples were collected in order to measure the viscosity, TAN, TBN and oxidation induction time. The output of the sensor initially decreased, most likely due to the

consumption or transformation of the additives in the oil [24]. The sensor output measured an increase in the oil conductivity after the addition of new oil, and also a further increase as the miles of driving increased, indicating the accumulation of acidic by-products from oil degradation. This increase in acidic by-product concentration was confirmed through TAN tests [24]. Since conductivity indicates the capability of a material to conduct an electrical current, this study suggests that there is a possible correlation between TAN and the dielectric constant. As TAN increases, due to accumulation of acidic by-products, the dielectric constant is most likely to increase.

These studies also indicate that the conductivity of the lubricant is affected by soot content. Both conductivity and dielectric constant are sensitive to soot contamination and nearly insensitive to fuel dilution. Viscosity may show no change over an operational period if there is both fuel dilution and soot contamination.

Discussions on the lubricant properties and their trends give indication to the need for direct, in-situ monitoring of multiple properties of engine oil. This approach would readily reveal trends that may show sudden spikes or drops in a property, indicating a change in lubricant condition or an engine problem that needs to be addressed.

1.8 Current Oil Monitoring Technologies and Strategies

Oil monitoring technologies are being investigated for ground vehicle use and for industrial applications. Some of the technologies currently being investigated are: quartz crystal microbalance (QCM) thickness mode systems; micro-acoustic wave technology;

mechanical resonators (i.e., tuning fork resonators); conductive polymer bead matrix technology; and infrared (IR) absorption technology (i.e., miniature spectrometers).

Research has been devoted to the development of a micro-electromechanical system (MEMS) multi-sensor array equipped with a quartz crystal microbalance (QCM) [25]. This sensor was designed for industrial hydraulic and lubricating systems. The sensor is able to simultaneously measure temperature, relative humidity, relative viscosity and changes in the dielectric constant. There is a sensing element for each of the physical properties on the substrate. Calibration is required for each type of fluid and operating condition.

The shift in resonant frequency of the QCM, as a result of damping from a fluid mass, is proportional to the liquid mass deposited on the QCM. The deposited liquid mass varies as the square root of the product of fluid density and viscosity. From these relationships, the viscosity values for the fluid are calculated.

A resistive temperature detector (RTD) is used for measuring the temperature. A capacitor, constructed with a polyimide between platinum electrodes, is used for measuring the relative humidity. Inter-digital capacitors are used for measuring the dielectric constant.

The sensor was tested in laboratory conditions in which fluid circulated at a rate of 5L/min. in a tank. Oil samples were taken and sent to a laboratory for TAN and viscosity measurements, using ASTM D 664 and ASTM D 445, respectively. These sensor measurements were compared to laboratory results.

The viscosity trend obtained from the sensor nearly overlapped the trend established with the viscosity values from the laboratory analysis. An exponential increase in

viscosity was observed, the increase being expected as a result of polymerization [25]. Dielectric constant measurements were plotted with TAN values against the time interval. The dielectric constant increased at the same rate as the TAN. The increase in both the dielectric constant and TAN was expected as a result of oxidation.

Any correlation established with these tests cannot be carried over to all hydraulic and lubricating oils. This is a result of the complexity of lubricating oils and differences between base stocks and additive packages. Nonetheless, the observed trends and the explanations for the occurrence of these trends can be extended to other lubricants.

In [26], the authors investigated the development of an oil condition sensor which measures viscosity, permittivity, and temperature. The sensor is installed at the bottom of the oil pan and sealed by a gasket. Experimental work involved tests with soot and fuel contaminated oil samples, since these are the most prevalent contaminants in diesel engine oils.

The viscosity was measured using a device on the sensor that uses micro acoustic wave technology. The output from this device is proportional to the inverse of the square root of the kinematic viscosity [26]. Two types of tests were conducted to measure the sensor response to fuel diluted samples. The first tests involved samples of new oil manually diluted with fuel. The second set of tests involved samples from diesel engine tests. For both tests, the sensor output showed a linear, decreasing trend as the fuel content increased.

While changes in viscosity reflect multiple oil degradation mechanisms, such as fuel dilution, soot contamination, and oxidation of the base stock, soot contamination appears to be the dominating cause for changes in dielectric constant. To test the sensor response

to soot contamination, oil samples were taken from a diesel test engine at regular intervals. The measured dielectric constant from the sensor showed an increasing, linear trend as the soot content increased [26].

Microacoustic technology has been a common technique to monitor oil condition as evidenced by its use from multiple developers of oil-condition sensors. In [27], the sensing element performs shear oscillations while in contact with the lubricant.

Accumulation of the lubricant on the sensing element leads to an exponential decrease in the distance traveled by the sensing element during the shear movement. The decrease in this distance can be expressed in terms of the liquid dynamic viscosity, density of the lubricant, and the angular frequency of the oscillation. The loading of the lubricant on the sensing element dampens the oscillation and changes the resonance frequency of the resonator. These effects are proportional to the square root of the frequency, dynamic viscosity, and density product.

An oil condition monitoring approach similar to the microacoustic method is the tuning fork resonator, as discussed in [28]. This technology enables measuring four independent physical parameters: dynamic viscosity, density, permittivity and electrical conductance. Electrodes are placed on the tuning fork, which elastically deforms due to an alternating voltage. As the tuning fork vibrates, an alternating current passes through the electrodes, enabling the electrical impedance (i.e., the ratio of the alternating voltage and alternating current) from this system to be calculated. The measured complex impedance is modeled as an equivalent electrical circuit which takes into account the mechanical, electrical and hydrodynamic effects of the lubricant on the tuning fork. It is reported that this system is not easily disturbed from vibrations, shocks or noise.

Conclusions from the investigation on the tuning fork technology revealed that the sensor outputs for viscosity in a bench-top laboratory setting and engine testing environment correlated well with the calculated viscosity, based on ASTM D 341-03, and oil analysis data. Also, the sensor electronics proved to be durable in all the fall, spring and winter seasons [28].

Another type of sensor evaluated in the literature review utilizes a conductive polymer matrix which monitors changes in the solvent properties of the oil to quantify oxidation, as well as water and antifreeze contamination [29]. This sensor consists of a nonconductive plastic strip with two orifices, each covered by a stainless steel screen which contains an insoluble polymeric matrix. The stainless steel screen serves as the electrodes for the sensor. Degraded engine oil is able to flow through one of the orifices, whereas the other one is sealed with clean oil, serving as a reference. The sensor extends from the drain plug in the oil pan and is connected to a signal conditioning unit. The polymer matrix has resin beads which have a charge associated with them and thus act as a conducting medium. The electrical properties of the polymeric matrix change as the charged resin beads adjust to the changing solvent properties of the oil. Clean oil is nonpolar; therefore the charged beads are bridged together, allowing for electrical transfer, measured in terms of resistance. As engine oil degrades its polarity increases, and as it flows through the stainless steel screen, through the polymer matrix, the resin beads become less efficient in transferring an electrical signal due to an increase in resistance. The increase in resistance is correlated with a change in solvent properties of oil [29].

Bench-top tests were conducted with used oils, unused oils, and oils contaminated with water. In addition, on-board testing was conducted in vehicles driven over the

course of an oil change. This involved city and highway driving, testing in old vehicles which required oil every 2000 miles and in new vehicles.

The following conclusions were drawn from these experiments: the sensor was able to track oxidation in real time; water contamination was monitored independently of oxidation; the sensor was able to operate properly over the temperature range experienced in a vehicle and it was insensitive to different oil types; there was a clear difference in sensor output between fresh and used oils; and results from complementary laboratory analysis (e.g., infrared spectroscopy, differential scanning calorimetry, thermogravimetric analysis, dielectric analysis, and metal analysis) correlated closely with the sensor output [23].

A unique oil-condition sensor under investigation uses an integrated infrared absorption sensor system; that is, a miniature spectrometer [29]. Such a device would allow for quantifying the extent of oil degradation based on oxidation as measured through infrared absorption at two particular wavelengths. Design considerations have been proposed but further research is necessary in order to produce a device with adequate sensitivity.

There are several oil monitoring strategies in current use. Many of these have been developed that use operational parameters, the electrical properties of the oil, and an algorithm to alert the vehicle operator on the condition of the oil. General Motors uses the Oil-Life System, which integrates feedback on engine revolutions, operating temperature, and other parameters with a computer algorithm to detect the need for oil changes [30]. This approach provides an indirect method to determine the oil change

interval. Daimler Chrysler's oil monitoring device monitors engine parameters as well and in addition, integrates a dielectric sensor to monitor the condition of the oil.

As previously discussed, oil monitoring strategies have been in existence for some time and there have been several approaches for monitoring oil quality. The most effective monitoring strategy is likely to be dependent on the application. To assist in this decision process for the case in which a fleet of vehicles are maintained, oil analysis programs are often established. The oil analysis program monitors both equipment and lubricant condition [31]. Such a program would be more efficient if the lubricant was analyzed in on-site laboratories. On-site analysis can be accomplished through an on-board monitoring sensor.

Research has been conducted on commercially available instruments and developmental technologies [31]. A summary of the findings in such research would be an exhaustive list of companies and their oil analysis instruments. More pertinent in regards to this paper are the means by which the research was conducted and the final recommendations. During the research, the instruments were evaluated based on the following parameters: weight size, durability, capabilities, power requirements, number of hours of self-sustained operation, and cost.

The results from this study indicated that there is a possibility, with good reason, to incorporate a commercial instrument or a device with multiple instruments to monitor the condition of the oil. This supports the significance of research on devices, such as the oil condition sensor used in this research, which are able to measure multiple parameters.

Table 7 summarizes the oil sensors previously discussed along with their working principles and measured parameters. As seen in Table 7, there are two main choices of

variables to measure: engine operating parameters or physical properties of the engine oil. The physical properties of the lubricant are a better choice because they provide a direct indication of oil condition. Measuring the engine operating parameters would overlook fuel dilution or other engine problems that might directly affect the lubricant.

| Oil Sensor | Working Principles | Measured Parameters |
|-------------------------|-----------------------------|-----------------------------------|
| Micro-electromechanical | Electromechanical system | Temperature, relative humidity, |
| system (MEMS) multi- | | relative viscosity and changes in |
| sensor array | | the dielectric constant |
| Conductive Polymer Bead | Polymer bead matrix | Solvent properties of lubricant |
| Matrix | | |
| Microacoustic Sensor | Microacoustic wave | Viscosity |
| | technology | |
| IR-Absorption Sensor | Miniature spectrometer | Oxidation |
| GM Oil-Life System | Algorithm | Engine operating parameters |
| Daimler Chrysler Assyst | Algorithm complemented | Engine operating parameters and |
| | by dielectric constant | dielectric constant |
| | measurement | |
| Tuning Fork mechanical | Impedance related to motion | Density, dynamic viscosity, |
| resonator | of sensor and physical | permittivity and electrical |
| | properties of fluid | conductance |

Table 7: Oil condition sensors

It is also beneficial to use a monitoring technology that operates on an in-situ, continuous basis, enabling ease of use in the field. Also, the technology should be able to monitor multiple properties, since oil degradation involves physical changes (indicated by changes in viscosity) and chemical changes (indicated by changes in the electrical properties of the oil).

The sensor that was used in this research measures multiple parameters: viscosity, temperature, density, and dielectric constant, using tuning fork resonator technology. It was selected based on its multi-parameter measuring capability, its availability, and

likelihood for commercial deployment. The sensor measures oil properties on a direct, insitu, and continuous basis. Table 7 compares the working principles and measured properties of the oil sensor used in this investigation with respect to other sensors on the market.

1.9 Thesis Outline

The research presented here was divided into two parts: bench-top experiments and engine experiments. Bench-top experiments were conducted to validate the accuracy and precision of the lubricant properties as measured by the oil-condition sensor prior to its installation in the engine and to establish correlations between contamination levels and changes in lubricant properties. Engine experiments were conducted to quantify changes in lubricant properties with respect to engine operating time, establish correlations between measured properties and determine potential causes for the property changes.

The remainder of this thesis is structured as follows: Chapter II discusses introductory information about the oil sensor and assesses the validity of the lubricant properties as measured by the sensor. Chapter III describes the experiments conducted on oil samples with a single contaminant and presents the results from these tests. Engine experiments are discussed in Chapter IV. Chapter V summarizes the research, presents conclusions and provides recommendations for future work.

CHAPTER 2

EXPERIMENTAL APPROACH

2.1 Working Principles and Description of Sensor

The oil-condition sensor is a solid state, piezoelectric tuning fork resonator. The tuning fork is made from quartz and is protected by a shroud. There are openings in the shroud allowing fluid to flow across the tuning fork, enabling measurements to be conducted. The shroud with the openings is evident in the Figure 2.



Figure 2: On-board oil-condition sensor

The sensor begins recording measurements once an AC voltage is applied to the electrodes. This creates an oscillating mechanical stress in the tuning fork. As the tuning fork vibrates, the electrical current, which is passed through the electrodes, changes. The ratio of the applied AC voltage and the resulting current yields the impedance of the tuning fork. This impedance has a dependence on the mechanical motion of the sensor, which in turn depends on the physical properties of the fluid. Therefore, changes in the physical properties of the lubricant can be quantified by measuring impedance changes as a function of input frequency. The theoretical model developed to calculate the impedance in terms of the physical properties of the fluid is shown in Equation 3 [32]:

$$Z(\omega) = Ai\omega\rho + B\sqrt{\omega\rho\eta}(i + 1)$$
 [Eq. 2.0]

Where:

i = current

 ω = operation frequency

 ρ = fluid density

A = coefficient depends on flow geometry only

 η = fluid viscosity

B = coefficient depends on flow geometry only

To ensure this theoretical model is accurately matched with the tuning fork response, the following conditions must be satisfied [33]:

- The oscillation velocity should be much less than the velocity of sound in the surrounding liquid.
- ii. The size of the resonator should be much smaller than the wavelength of compression waves in surrounding liquid.
- iii. The size of the resonator should be much bigger than the thickness of boundary layer.
- iv. The surface of the resonator should be smooth; the roughness should be much smaller than the thickness of a boundary layer.
- v. The oscillation amplitude should be much less than the thickness of the boundary layer.

The sensor simultaneously and directly measures the lubricant temperature, dynamic viscosity, density, and dielectric constant on a real-time, in-situ basis. The temperature range over which each sensor component is able to properly operate is shown in Table 8. The limiting operating condition through which property measurements can accurately be made are shown in Table 9.

| Sensor Component | Continuous Operating Temperatures |
|----------------------------------|--|
| SCU Box | -40 to +85°C |
| Sensor Electronics | -40 to +125°C |
| Sensor Element Fluid Temperature | -50 to +150°C |
| Cabling (FEP insulated) | -70 to +200°C |

Table 8: Operating temperature limits for sensor components [34]

| Physical Properties of Fluid | Continuous Operating Conditions |
|------------------------------|--|
| Dynamic Viscosity | 0.0 to > 50.0 cP |
| Density | 0.000 to 1.500 g/cm ³ |
| Dielectric Constant | 1.00 to 6.00 |
| Fluid Temperature | 55°C to +150°C |

Table 9: Limiting operating conditions for sensor [34]

2.2 The On-Board Oil-Condition Sensor Testing Platform

The oil condition sensor connects to a sensor control unit (SCU). The SCU has a standard RS-232 Com port by which it interfaces with a host computer via a communications cable. Input power to the SCU is provided via a 12V power supply. This set-up is shown in Figure 3. All of these components used to operate the sensor are referred to as the Development Test Platform (DTP). The data from the sensor are recorded to a text file and displayed on the host computer using ASIC Studio, proprietary software developed by the sensor manufacturer.

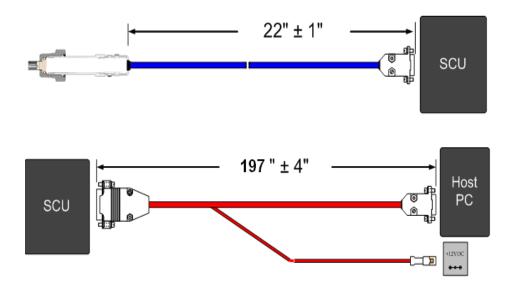


Figure 3: On-board oil condition sensor set-up [34]

a. Target Applications

The sensor was designed to measure the physical properties of various fluids, such as engine oils, transmission oils, hydraulic oils, corn and organic oils, diesel fuels, kerosene, and refrigerants. From an engine perspective, the ideal installation is near the inlet or outlet of the oil filter head or engine block gallery. The sensor is able to perform well under flow rates ranging from 0.2 m/s to 10 m/s. Flow conditions are preferred over stagnant conditions. The sensor can withstand pressures between 0 psi and 350 psi [39]. Figure 4 shows possible orientations for mounting the sensor in an engine. Figure 5 shows possible orientations for mounting during bench-top testing.

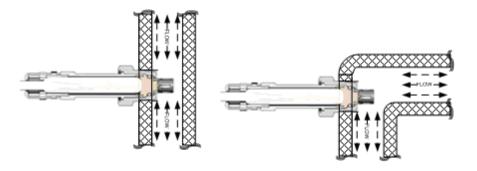


Figure 4: Sensor orientation for engine mounting [34]

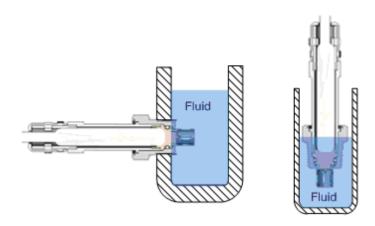


Figure 5: Sensor orientation for bench-top testing [34]

b. Validation of lubricant properties measured by oil-condition sensor

The validity of the lubricant properties measured by the sensor (i.e., objective 1) was determined by quantifying the accuracy and precision of the property measurements through a series of comparative, bench-top experiments. The validity of the temperature was assessed with a thermometer. The kinematic viscosity (i.e., the ratio of dynamic viscosity and density) was validated by comparing sensor readings to results obtained from a method developed by the American Society of Testing and Materials (ASTM), Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids and Calculation of Dynamic Viscosity (ASTM D

445) [15]. Since no standardized methods for dielectric constant measurements are available, a calibrated bench-top sensor (Brookhaven's BI-870 Dielectric Constant Meter) was used to validate the dielectric constant output. The validity of the BI-870 dielectric constant meter was also verified by comparing the dielectric constant values measured with the instrument to values obtained from fluids with known dielectric constant.

2.3 Bench-Top Experiments For Sensor Output Validation

Two experimental set-ups were used for bench-top experiments involving the on-board oil-condition sensor. The first set-up, suggested by the instrument manufacturer, is shown in Figure 6. Engine oil was poured into an 800mL beaker. A stir bar was introduced into the beaker, which was placed on a hot plate. The sensor and a thermometer were placed in the engine oil, with the sensor at least two-and-a-half inches above the stir bar. The thermometer was at the same depth as the sensor. In this set-up, the oil was flowing in a circular path, passing through the shroud and across the sensing element of the oil-condition sensor.



Figure 6: Experimental set-up 1

In the second set-up, shown in Figure 7, mineral oil was poured into a 1000mL beaker. A stir bar was introduced into the beaker, which was placed on a hot plate. Engine oil was poured into a glass container. This container was held in the mineral oil at least two-and-a-half inches above the stir bar. The sensor was placed in the engine oil. A thermometer was placed in both the mineral oil and engine oil, each at the same depth as the sensor. This set-up was based on the idea that it would produce a uniform temperature distribution around the engine oil, while using less engine oil. By comparing the set-ups, it was possible to determine if the accuracy and precision of the sensor output is affected by whether the fluid is moving or static. Two types of engine oils were used in both experimental set-ups for the bench-top experiments so that any dependence of the measured properties on the oil type could be assessed. The lubricants were: Motorcraft® 15W-40 Super Duty Diesel Motor Oil and Rotella® T SAE 15W-40 with advanced soot control. The experimental setups just described will be subsequently referred to as Experimental Set-ups 1 and 2, respectively.



Figure 7: Experimental set-up 2

Once the sensor was placed in the engine oil, the hot plate and the stirrer were turned on and the sensor operation was started. During the tests, the temperature of the oil was

increased continuously beyond 100°C to allow the additive package to activate [40] and was stabilized for 5 minutes at approximately 112°C. After this time, the hot plate was turned off and the oil cooled to ambient temperature. Measurements were continuously acquired with the oil-condition sensor during the heating, stabilization and cooling processes. Temperature readings from the thermometer were simultaneously recorded.

The sensor records a temperature at the beginning of the measurement (referred to as the initial media temperature) and a temperature at the end of the measurement (i.e., the final media temperature). One complete measurement lasts just over one minute. As the oil-condition sensor operates, the interface from the ASIC software shows the impedance as a function of time. Upon completion of a measurement the lubricant properties are displayed. Measured temperatures and property values are recorded in a text file. The average media temperatures, calculated using the initial and final media temperatures, were used as reference temperatures when analyzing the property values. It is known that the sensor output is not reliable when the difference between the final and initial media temperature is greater than one degree. Lubricant properties at those temperatures were neglected to ensure measurement accuracy.

2.4 Data Processing

It was observed that extraneous density and viscosity values were recorded by the sensor below 55°C regardless of experimental set-ups and oil type. This indicates that the lubricant property measurements are inaccurate below this temperature. Upon inspection of the data, the differences between the initial and final media temperature was less than one degree, indicating that the data should be valid.

Table 10 displays the range for which kinematic viscosity (i.e., ratio of the dynamic viscosity and density) values were measured by the oil sensor and the expected range for the kinematic viscosity at the stated temperature range.

| Lubricant Temperature Range (°C) | Kinematic Viscosity Range as Measured by the On-board Sensor (cSt) | Expected Kinematic Viscosity Range (cSt) |
|----------------------------------|--|--|
| 21-54 | 1-48 | 344-65 |

Table 10: Extraneous kinematic viscosity range measured by the on-board sensor

For a temperature of approximately 21°C, the on-board sensor recorded a kinematic viscosity of approximately 1 cSt. Likewise, the on-board sensor recorded approximately 48 cSt for an approximate temperature of 54°C. The uniqueness of this data is that the kinematic viscosity values actually increase as the temperature increases; this is contradictory to the expected trend. The kinematic viscosity decreases as the temperature increases for unused oil, as was used in these experiments. Contamination may cause the oil to show a trend different from the norm.

In order to assess the error in the magnitude of these recorded kinematic viscosity values ASTM D 341: Standard Test Method for Viscosity-Temperature Charts for Liquid Petroleum Products was used [15]. This standard provides equations by which extrapolated and interpolated kinematic viscosity values at a given temperature can be calculated provided that two kinematic viscosity values at given temperatures are known. The kinematic viscosities at 40°C and 100°C, as taken from the Shell Material Safety Data Sheet (MSDS), were used in ASTM D 341 to calculate the expected kinematic viscosity at 21°C and 54°C. These values, as shown in Table 10, are 344 cSt and 65 cSt, respectively. The expected kinematic viscosity range exhibits the actual trend in

viscosity for an increasing temperature range for the case of unused oil. In addition, the expected, kinematic viscosity at approximately 21°C: 344 cSt, is two orders of magnitude greater than the kinematic viscosity as recorded by the sensor at approximately 21°C. Regardless of oil type and experimental set-up, extraneous viscosity and density values were continuously recorded for this temperature range. This analysis shows that the lubricant property values are not reliable for a temperature range of 21-54°C.

Another characteristic of the data recorded to the text file was the occurrence of inflection points within this temperature range in the dynamic viscosity data. Whereas the extraneous values consistently occurred over the same range of operating temperatures for two different experimental set-ups and two different engine oils, the inflection point occurred only twice for a single experimental set-up and engine oil. The inflection points are further evidence of the measured lubricant properties not being accurate within this temperature range.

While these findings serve to establish the lower limit for the accuracy of the lubricant property measurements, it does not impose any limitations for measuring lubricant properties in an engine, where temperatures above 55°C can be expected under normal operating conditions.

The following sections assess the accuracy and precision of the measured temperature, kinematic viscosity, density, and dielectric constant.

2.5 Precision and Accuracy Assessments

a. Temperature

For all bench-top experiments using the on-board oil-condition sensor, a thermometer was placed in the testing beaker. The thermometer reading was recorded at the completion of each measurement from the oil-condition sensor. The following graphs show the temperature as measured from the thermometer and sensor for the duration of the test. Figure 8 corresponds to experimental set-up 1 using Shell engine oil. The thermal cycle of the test can easily be distinguished in the figure.

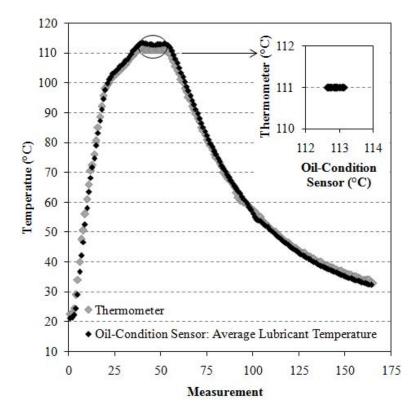


Figure 8: Experiment 1 for Shell engine oil using experimental set-up 1

As the temperature increased at a rate of three to five degrees per minute, the sensor output lagged the thermometer reading by approximately four degrees. Once the temperature stabilized, according to the thermometer measurement, the sensor

measured a temperature no less than a degree from the thermometer reading. This is shown in the inset on Figure 8. Once the temperature began to decrease, the sensor temperature values were approximately a degree less than the thermometer measurements. This suggests that the accuracy of the temperature measurement improves the longer the sensor is exposed to the operating environment, which points to a limit on the response time of the sensor and suggests that it must be allowed to reach thermal equilibrium with the environment to obtain accurate property measurements. Figure 9 illustrates temperature measurements from the on-board oil condition sensor for a second experiment using experimental set-up 1 and Shell engine oil.

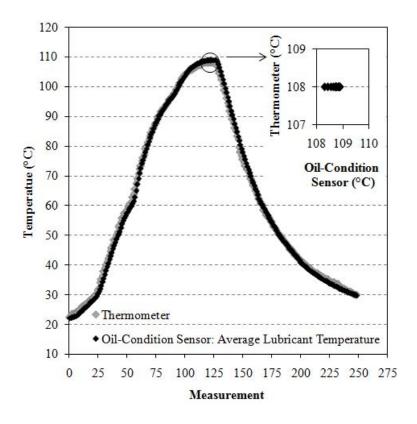


Figure 9: Experiment 2 for Shell engine oil for experimental set-up 1

During this experiment, the rate of temperature rise was set to one to two degrees per minute, which is lower than for the previous experiment. This resulted in a smaller difference between the sensor and thermometer measurement during the heating period, one to two degrees versus four degrees. The difference between the sensor and thermometer measurement during the stabilization period and the cooling period were the same as they were for experiment one, approximately a degree.

Similar temperature plots (not shown to avoid redundancy) were obtained from additional tests using Motorcraft and Shell engine oils. These graphs confirmed the findings just summarized from Figures 8 and 9: that the sensor needs at least twenty minutes to reach thermal equilibrium in order to deliver accurate outputs. This does not present a problem for the target application, as the sensor will be used for steady state measurements in engines. As expected, the temperature measurements are not affected by experimental set-up. Results support the accuracy and precision of the temperature measurements in both a moving and static fluid once thermal equilibrium is reached.

b. Kinematic Viscosity

The kinematic viscosity, as calculated from the ratio of the dynamic viscosity and density, was plotted for both experimental set-ups and engine oils. The kinematic viscosity values for the duration of the thermal cycle for Shell engine oil are shown on a single graph, Figure 10. In addition, the experimental kinematic viscosity at 100°C as measured according to ASTM D 445 is shown.

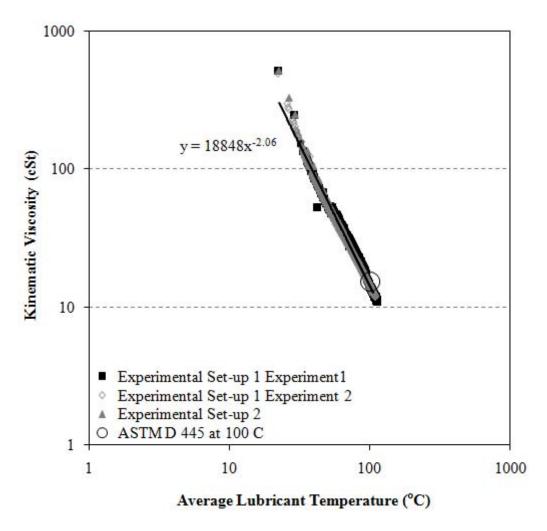


Figure 10: ASTM viscosity measurement plotted against sensor measurements for a thermal cycle using Shell engine oil

The viscosity values as measured by the sensor show a power law decrease, as indicated by the trend line. In addition, the viscosity values as measured by the sensor overlap the average viscosity value from the ASTM method measured at 100 °C. Although the viscosity values at 100 °C from the sensor overlap the viscosity value from the ASTM method at this temperature, it would be more useful to have exact values from the sensor to compare to the viscosity measurements from the ASTM method. Since the sensor was operating in a transient environment the kinematic viscosity values during the stabilization period were used in ASTM D 341

to calculate what the viscosity would be as measured by the sensor at 100°C. This was the same standard used in evaluating the extraneous viscosity measurements from the sensor. Since viscosity has a dependence on temperature, it is imperative to reference the temperature at which the viscosity was measured. Viscosity values from the sensor stabilized at a temperature higher than 100°C, approximately 112°C. The lower and higher viscosity value, at which the temperature was stabilized were used in ASTM D 341 to calculate what the viscosity would be, as measured by the on-board sensor, at 100°C. This value was compared to the average viscosity measured according to ASTM D 445 at 100°C.

Figure 11 shows the average kinematic viscosity measured according to ASTM D 445 (i.e., bench-top experiment), and the kinematic viscosity value for all experimental set-ups measured with the on-board sensor and adjusted using ASTM D 341. All viscosity values in figure 2.9 are referenced to 100°C.

Figure 11 shows that the experimental set-up had a small effect, if any on the sensor's capability to precisely measure the viscosity. The standard deviation (i.e., precision) between the viscosity measurements from the on-board sensor is ± 0.1 . The viscosity values measured from ASTM D 445 show the same precision as the measurements from the on-board sensor. The discrepancy between the sensor output and results from ASTM D 445 is 5.3%. This is less than the change in kinematic viscosity that is considered a condemning limit (i.e., a change of 10-18 cSt or a change of $\pm 0.18\%$). A summary of this discussion is shown in the following table. The actual values for the data points shown in Figure 11 are displayed in Table 11.

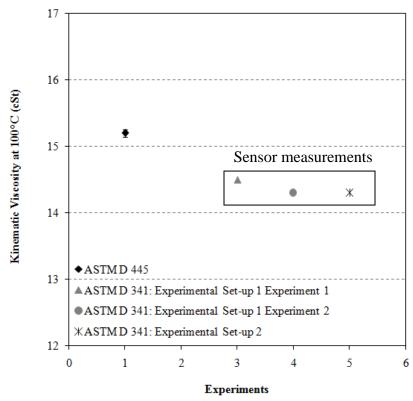


Figure 11: Comparison between experimentally determined kinematic viscosity values

| | ASTM D 341 | | |
|------------|---------------------|---------------------|----------|
| ASTM D 445 | Set-up 1 Experiment | Set-up 1 Experiment | Set-up 2 |
| | 1 | 2 | Set-up 2 |
| 15.2 cSt | 14.5 cSt | 14.3 cSt | 14.3 cSt |

Table 11: Viscosity values from ASTM D 445 and sensor (ASTM D 341)

The kinematic viscosity values calculated from ASTM D 341 using the sensor data show both precision and accuracy, supporting the oil-condition sensor's capability to measure the viscosity at typical engine operating temperatures.

Figure 12 shows the viscosity trend for Motorcraft engine oil for the two experimental set-ups discussed in Section 2.3 of Chapter 2. This graph shows a similar trend to the Shell engine oil (i.e., a power law decrease in the viscosity),

further confirming the sensor capability to measure viscosity over a temperature range typical of IC engine operation independently of oil type.

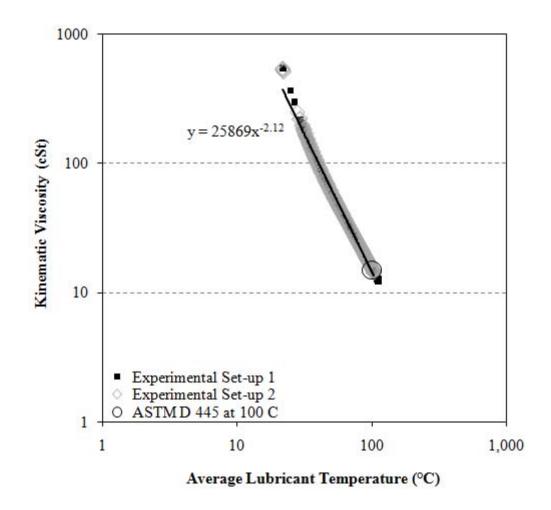


Figure 12: ASTM viscosity measurement plotted against sensor measurements for a thermal cycle using Motorcraft engine oil

c. Density

The density measured with the on-board sensor was not compared to other experimental density values. An analysis of the density trend over the thermal cycle was conducted since the density was used to calculate the kinematic viscosity values shown in the previous section. In addition to the density having relevance in the

calculation of the kinematic viscosity, it is significant in the calculation of the dielectric constant, as previously shown in the Debye equation in Chapter I. Figure 13 shows the trend in the density during a thermal cycle for both experimental set-ups using Shell engine oil.

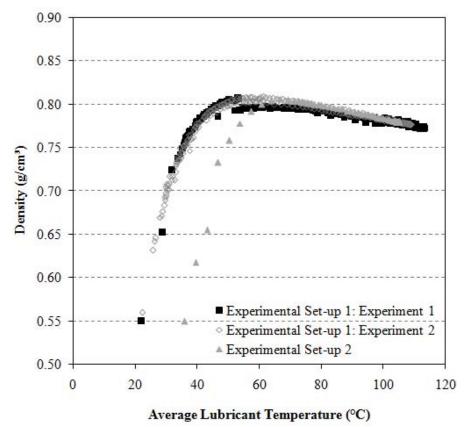


Figure 13: Density measurements from sensor during a thermal cycle for both experimental set-ups using Shell engine oil

Figure 13 shows the density initially increasing as the temperature increases and then eventually decreasing as the temperature continues to increase. Density is inversely related to the temperature. Therefore, as the temperature increases the density should decrease, not increase as is initially observed in Figure 13. Figure 14

shows the trend of the density measurements for the experimental set-ups using Motorcraft engine oil.

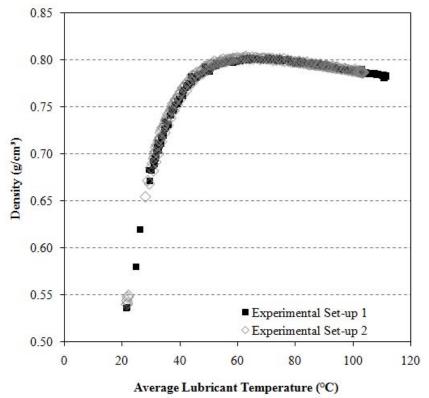


Figure 14: Density measurements from sensor during a thermal cycle for both experimental set-ups using Motorcraft engine oil

The trend in the density observed with the Shell engine oil is also observed with the Motorcraft engine oil for both experimental set-ups, as shown in Figure 14. This indicates that the density trend is sensor-related and not dependent on oil type. It further indicates the sensor does not give accurate measurements at temperatures below approximately 55°C, as discussed in Section 2.5 of Chapter 2.

d. Dielectric Constant

The dielectric constant was the most difficult property to validate. As previously discussed, the trend in dielectric constant over a temperature range is dependent on the base oil, additives, oil degradation, and contamination. Further yet, as mentioned in Chapter 1, it has been reported that the operating frequency of the sensor can have an impact on the measured dielectric constant. This makes it difficult in assessing the differences in the dielectric constant as measured from two sensors operating on different frequencies.

Despite these challenges, an effort was made to assess the accuracy and precision of the on-board sensor. At the very least, if there is an order of magnitude difference in the measurements from two sensors then there should be concern in the capability of the sensor to accurately and precisely measure the dielectric constant.

Measurements of the dielectric constant from the oil-condition sensor were compared to measurements taken from a bench-top dielectric constant meter (Brookhaven Instruments' BI 870). It has an operating range between 22°C and 58°C [35]. It has already been shown that the on-board sensor outputs for viscosity and density are unreliable below 54°C. However, mere inspection of the dielectric constant data below 54°C does not give any indication of data reliability because the measured dielectric constant values are well within range of the expected dielectric constant for engine oils. For this reason, the sensor output was tested over a broad temperature range.

The output from the BI-870 sensor was validated before using the instrument to assess validity of the on-board oil condition sensor readings. This was accomplished

by conducting tests on methanol, de-ionized water, chlorobenzene, and cyclohexane.

These liquids have a known dielectric constant at ambient temperature. Results from these tests are shown in Figures 15 and 16.

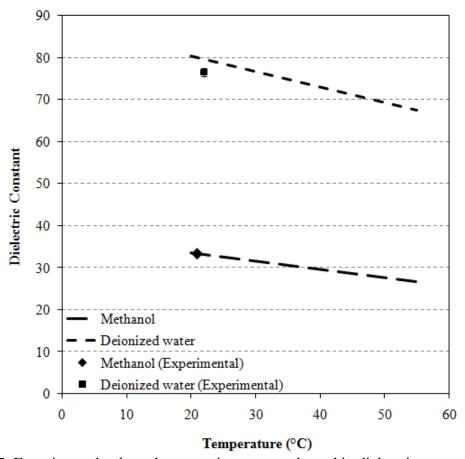


Figure 15: Experimental values shown against expected trend in dielectric constant with a change in temperature for methanol and de-ionized water

Figure 15 shows that the dielectric constant measurement for methanol from the Brookhaven instrument intersects the expected values for methanol. Good agreement is found between the predicted and measured dielectric constant values for de-ionized water. The discrepancy between the predicted and measured value for de-ionized water is approximately 4%. The standard deviation for a set of three measurements

was 0.06 for methanol and 1.03 for the de-ionized water. Measurements for both methanol and de-ionized water show good precision, although slightly better for the methanol as indicated by a lower standard deviation.

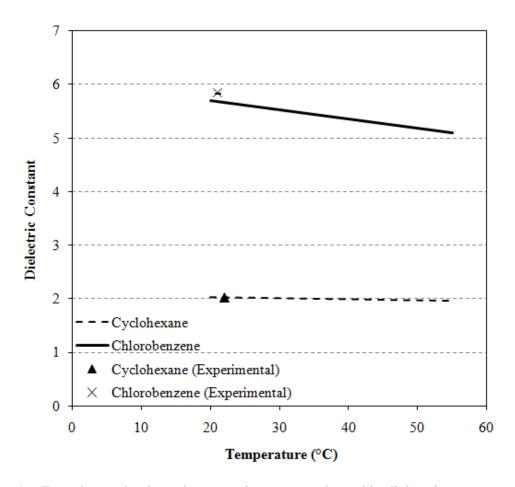


Figure 16: Experimental values shown against expected trend in dielectric constant with a change in temperature for cyclohexane and chlorobenzene

As shown in Figure 16, the dielectric constant of cyclohexane and chlorobenzene measured with the BI-870 instrument are in good agreement with the expected values. The discrepancy between the predicted and measured value for chlorobenzene is approximately 2%. Good precision was achieved for both sets of measurements. The standard deviation for a set of three measurements was 0.01 for both cyclohexane and

chlorobenzene. Since good precision and accuracy were obtained when validating the BI-870 dielectric constant meter, this instrument was used to assess the precision and accuracy of the on-board sensor.

Figure 17 shows the trend observed for both the on-board sensor and the BI-870 dielectric constant meter at stabilized temperatures. Verifying the output of the sensor over a temperature range previously deemed inaccurate for density and viscosity measurements is warranted by the fact that the dielectric constant measurements in this temperature range are within the expected values for engine oils (i.e, of 2.1 to 2.8).

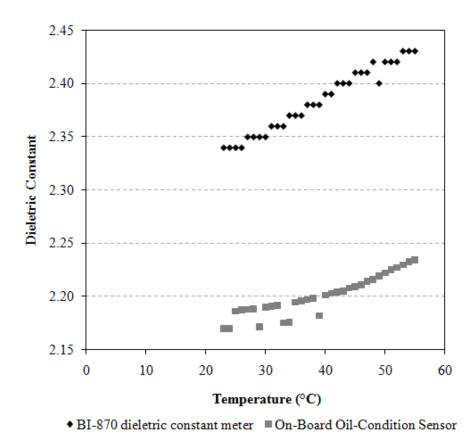


Figure 17: Dielectric constant as measured from BI-870 and oil sensor at stabilized temperatures for Shell engine oil

Both sensors show the trend of increasing dielectric constant with temperature. The measurements from the on-board oil-condition sensor are approximately 6.5 % less than the measurements from the Brookhaven instrument. As mentioned in Chapter 1 the dielectric constant can show frequency dependence if the measuring frequency of the instrument is above 106 Hz, as is the case with both instruments. It is hypothesized that this was the reason for the bias shown in Figure 17.

Figure 18, shows the dielectric constant as measured with the on-board oil-condition sensor during a thermal cycle. Two experiments were conducted using experimental set-up 1. After the first experiment, as denoted by Day 1, the beaker of oil was stored overnight and used the following day under the same testing conditions. The purpose in conducting a second experiment the following day was to check for permanent changes in the dielectric constant.

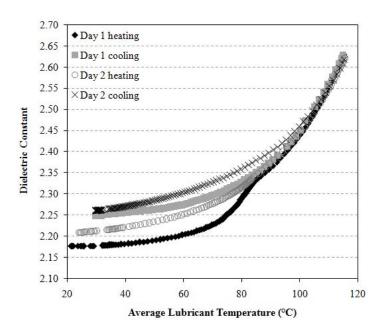


Figure 18: Dielectric constant during thermal cycle for experimental set-up 1 using Shell engine oil

Figure 18 shows the dielectric constant to increase as the temperature increases and then decrease as the temperature decreases for both days of testing. For each of the four tests shown in Figure 18, the dielectric constant is the same for temperatures 100°C and greater. At temperatures less than 100°C, during the cooling period, for both Day 1 and Day 2, the dielectric constant is greater than the corresponding dielectric constant at that given temperature for the heating phase. As the temperature increases the additives become active, modifying the chemical characteristics of the oil base stock. This process continues throughout the temperature range encountered in the thermal cycle. Figure 19 shows the dielectric constant trend for both experimental set-ups using the Motorcraft engine oil during a thermal cycle.

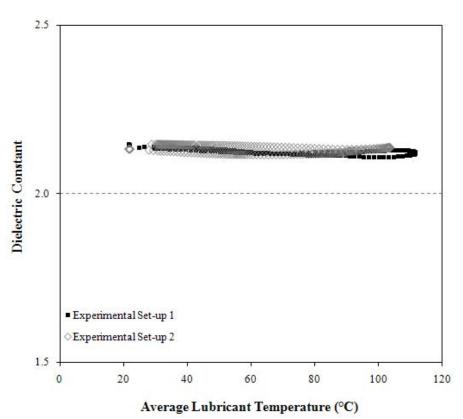


Figure 19: Dielectric constant during thermal cycle for both experimental set-ups using Motorcraft engine oil

In contrast to the Shell oil the dielectric constant of the Motorcraft engine oil shows little change as the temperature changes. This striking difference in the dielectric constant trend during a thermal cycle between the two oils tested (compare Figures 18 and 19) suggests a dependence of dielectric constant on oil type. This is not surprising, since it is known that the viscosity, paraffin/naphthene content, and the additive package contribute to the dielectric constant (see Chapter 1). Another noticeable difference between Figures 18 and 19 is the more prominent shift in the dielectric constant measurements for the Shell engine oil between the heating and cooling phases.

This shift in the dielectric constant measurements observed for the Shell engine oil was further investigated by analyzing the rate of temperature rise, shown in Figure 20. Since the experiments from both days have nearly the same rate of temperature rise, differences in the rate of temperature rise are not likely to account for the change dielectric constant measurements noticed between Day 1 and Day 2 (Figure 18). Therefore it is likely that the shift in the dielectric constant measurements is a result of permanent changes in the dielectric constant as a result of the oil undergoing a thermal cycle during Day 1.

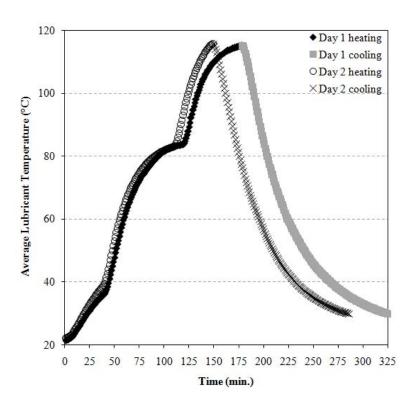


Figure 20: Rate of temperature increase and decrease for experiments using Shell engine oil

Figure 21 shows there is a difference in rate of temperature rise during the heating phase of the thermal cycle for Motorcraft oil. Since the dielectric constant trends for the Mortorcraft engine oil show virtually no difference despite the different rates of temperature rise (see Figure 19), this further supports the dependence of the dielectric constant on oil type.

In summary, the observed trends in the dielectric constant for both oil types are substantially different. Shell engine oil shows an increasing trend as the temperature increases and a decreasing trend as the temperature decreases. The dielectric constant of Motorcraft engine oil, on the contrary, remained constant during the thermal cycle. A plausible explanation is the different response of the additive package to temperature changes.

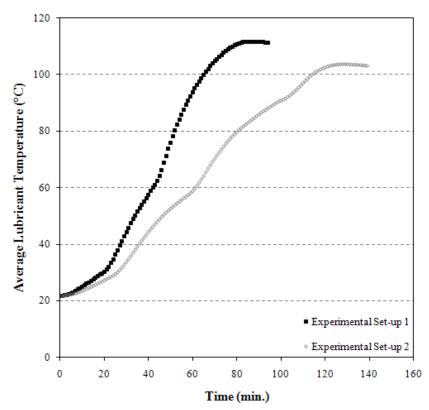


Figure 21: Rate of temperature increase for experiments using Motorcraft engine oil

CHAPTER III

SINGLE-CONTAMINANT EXPERIMENTS

3.1 Description of Single-Contaminant Experiments

Single-contaminant experiments were conducted in order to accomplish the second objective of this project: to establish correlations between the contamination level of a contaminant and the change in a given lubricant property. Prior to conducting measurements of samples with a single contaminant, a reference value needed to be established. This was accomplished through baseline measurements of the engine oil. Upon completion of the baseline measurements, experiments using oil samples with a single contaminant were conducted and regression analysis was used to draw conclusions. Property trends for these samples, correlations between percent of contamination and the change in properties, and the amount of contamination that resulted in a property reaching a condemning limit were assessed. The single contaminant that was considered was diesel fuel. This contaminant was selected because of its prevalence in compression ignition engines due to its low volatility.

3.2 Baseline Measurements

Baseline measurements were conducted to set the standard to which measurements from contaminated oil samples were compared to in order to quantify the extent to which a physical property had changed due to contamination. The MSDS for Shell Rotella® T Multigrade with Advanced Soot Control [36] includes the following property values:

kinematic viscosity (at 40°C and 100°C), as measured according to ASTM D 445; flash point, as measured according to ASTM D 92; and total base number (TBN), as measured according to ASTM D 2896. While taken as a general guide, these values were not used as the baseline. This is because for the same type of unused oil, properties differ depending upon the batch from which the oil originated and it was not possible to obtain the property values for the batch of oil used in the experiments. In order to limit the variance in properties between different batches, all samples were obtained from a single, fifty-five gallon barrel of oil. The barrel was kept indoors to minimize weather conditions initiating changes in the oil properties. Baseline measurements were conducted for the following properties: kinematic viscosity at 100°C and 40°C according to ASTM D 445; flash point according to ASTM D 92; TBN according to ASTM D 4739; and dielectric constant with the BI-870 dielectric constant meter. Table 12 shows a summary of the properties and their respective test methods for the baseline measurements.

| Bench-top Measurement | Test Method/Instrument |
|---------------------------------------|----------------------------------|
| Kinematic Viscosity at 100°C and 40°C | ASTM D 445 |
| Flash Point | ASTM D 92 |
| Base Number | ASTM D 4739 |
| Dielectric Constant | BI-870 Dielectric Constant Meter |

Table 12: List of properties and their test methods for baseline measurements

a. Kinematic Viscosity at 100°C and 40°C

According to ASTM D 445, a viscometer is used to measure the kinematic viscosity of lubricants. The type and size of viscometer is specified in the standard. The viscometer is filled with a specified amount of lubricant, approximately twelve milliliters, by inverting it into a beaker filled with lubricant and applying suction to

the other end of the tube. Engine oil is pulled through the tube to a mark on the viscometer. The viscometer is turned right side up and a plug is placed in the viscometer to prevent the lubricant from flowing into the timing bulb. The filled viscometer is placed in the temperature bath for at least thirty minutes in order to reach thermal equilibrium. After thirty minutes, the plug is removed and the time for the lubricant to flow through the timing bulb is measured. This time is multiplied by a calibration constant, specified for each viscometer, to calculate the kinematic viscosity (cSt). Table 13 shows the baseline measurement for the kinematic viscosity at 100° C and 40° C.

| | Baseline | Shell MSDS | Percent |
|--------------------------|-----------------|-------------|------------|
| | Measurement | Sileii MSDS | Difference |
| Viscosity at 100°C (cSt) | 15.2 ± 0.1 | 15.7 | 3% |
| Viscosity at 40°C (cSt) | 123.2 ± 0.2 | 118 | 4.4% |

Table 13: Kinematic viscosities for Shell 15W-40 diesel engine oil

b. Flash Point

The flash point was measured according to ASTM D 92. This method is often referred to as the Cleveland Open Cup method. A cup is filled with the test sample and then placed on a Bunsen burner. The heat input is adjusted according to the ASTM standard as the temperature is monitored. The flash point is recorded as the temperature at which sweeping a flame over the sample produces a flash over the entirety of the test sample. This temperature is corrected due to variances in the ambient pressure of laboratories. Table 14 shows results from the Flash Point test.

| Average, Corrected Flash Point | Shell Product Data Sheet |
|--------------------------------|--------------------------|
| 215 ± 2.8 °C | 213 °C |

Table 14: Flash point for Shell 15W-40 diesel engine oil

The error with this test is larger than the error in the kinematic viscosity tests.

One of the reasons for the larger error is the sensitivity of the measurement to the Bunsen burner control. Nevertheless, good precision and accuracy were obtained for both tests.

c. Total Base Number (TBN)

Since the majority of the experiments were conducted on used or contaminated oil samples, TBN measurements were conducted according to ASTM D 4739. This method was chosen instead ASTM D 2896 because it is more appropriate for used or contaminated oil samples, as discussed in Chapter I. Also, it was more cost effective to use a single test method, since ASTM D 2896 and 4739 use different chemical solutions for the titration.

The first step in this test method is to measure the potential of the pH 3 buffer. The potential of the pH 3 buffer and 100 mV + potential of pH 3 buffer need to be recorded. The next step is to measure the mass of the sample. The mass of the sample is calculated as follows [17]:

$$M = \frac{7}{E}$$
 [Eq. 3.0]

Where:

E = the expected TBN, reported on the oil's product data sheet

Following measurement of the amount of sample, the titration solvent is poured into the beaker. A stir bar is placed in the beaker and the beaker is placed on a plate with a magnetic stirrer. The electrodes are placed in the beaker. The titrant (0.1M) HCl solution) is added in 0.1 mL increments every ninety seconds, after which a potential reading is taken with the electrodes. This process continues until potential readings reach or surpass 100 mV pass the pH 3 buffer potential. The potential readings are plotted against the respective amount of titrant that was added, as shown in Figure 3.0. The significant point on the graph is the inflection point. The inflection point, that is the point where the sign of the curvature changes (i.e. the curve changes from being convex to concave or vice versa), is used to determine the end point. The end point is where all of the basic constituents are neutralized due to the addition of the acidic titrant. If the inflection point occurs at a potential that is between the buffer potential and 100 mV past the buffer potential, then the potential of the inflection point is treated as the end point. The volume of titrant corresponding to this end point is used in calculating the TBN. If the inflection point occurs below the buffer potential (as shown in Figure 22) or there is no inflection point, then the intersection between the titration curve and the buffer potential is taken as the end point. The final step prior to calculating the TBN is conducting a blank titration. A blank titration involves adding the titrant in 0.01 mL increments every twelve seconds to the titration solvent until the potential corresponding to the end point for the sample is reached. The purpose for conducting a blank titration is to determine if the titration solvent contributes to the basicity as measured from titrating the sample. If the initial potential of the titration solvent is greater than the recorded potential of the

end point for the oil sample, then the titration solvent does not contribute to the bascisitiy of the oil sample. The TBN is calculated as follows [17]:

$$TBN = \frac{(A - B) \cdot 0.1 \cdot 56.1}{m}$$
 [Eq. 3.1]

Where:

A = mL of titrant to reach the end point for the sample

B = mL of titrant to reach the end point for the blank

m = mass of sample

0.1 = molarity of the titrant

56.1 = constant specified by the ASTM standard

The results for the TBN tests are shown in Figure 22 and Table 15.

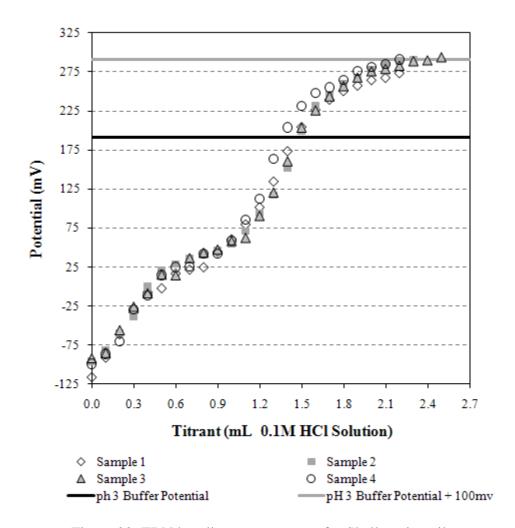


Figure 22: TBN baseline measurement for Shell engine oil

Figure 22 shows the titration curve for four measurements. The black line corresponds to the pH 3 buffer potential. The gray line corresponds to the pH 3 buffer potential plus 100mV. As seen in Figure 22, the inflection point in the titration curve occurs at a potential less than the pH 3 buffer potential. For this reason the end point was taken as the milliliters of titrant needed to reach the pH 3 buffer potential.

| Average TBN (ASTM D 4739) | Shell MSDS (ASTM D 2896) |
|---------------------------|--------------------------|
| 12.4 ± 0.4 | 11.5 |

Table 15: Comparison between experimental and reported TBN values

The average TBN baseline measurement for Shell engine oil is shown in Table 15 along with the TBN value from the Shell MSDS. It is important to emphasize that caution should be followed when comparing these values, since different test methods were used to conduct these measurements.

d. Dielectric Constant

The dielectric constant measurements obtained from the on-board oil-condition sensor and the BI-870 dielectric constant meter have previously been shown in Section 2.5d of Chapter 2. As previously discussed, both sensors show the trend of increasing dielectric constant with temperature. The measurements from the on-board oil condition sensor are approximately 6.5 % less than the measurements from the Brookhaven instrument. As mentioned in Chapter 1 the dielectric constant can show frequency dependence if the measuring frequency of the instrument is above 106 Hz, as is the case with both instruments. It is hypothesized that this was the reason for the bias.

3.3 Experiments with Fuel Contaminated Oil Samples

This section describes experiments conducted with unused oil samples manually contaminated with a known amount of fuel. All unused oil samples came from the same fifty-five gallon barrel from which oil was obtained for the baseline measurements. The fuel was obtained from a fuel container filled at a local Shell gas station with ultra low

sulfur diesel fuel. Fuel was added to the oil samples on a volume basis. These experiments addressed the following previously mentioned objectives:

- 1) Validate the accuracy and precision of the properties measured by the on-board sensor. Although this was accomplished for unused oil samples (see Chapter 2), the question of how accurate and precise the sensor measurements are when operating in a fuel contaminated environment is now being addressed.
- 2) Verify published correlations between fuel contamination levels and changes in lubricant properties. By fulfilling this objective, it was possible to determine the amount of fuel contamination that results in a physical property changing to its maximum or minimum limit, as specified in the literature.

The first step in proceeding with these experiments was to decide on the fuel contamination levels to test. Table 16 lists allowable limits for fuel contamination [5].

| Contaminant | Allowable Limits | Allowable Limits | Allowable Limits |
|-------------|------------------|--------------------|------------------|
| | | Minor 1.5-4.5% | 1 |
| Fuel | 0.5-1.5% by | Significant 5-7.5% | 2.5% to 5% max |
| | | Excessive >7.5% | |

Table 16: Fuel contamination limits by volume [5]

These limits are not specified for an engine, fuel, or oil type. For this reason, they were used as mere guidelines in selecting the fuel contamination levels. The following sections discuss the change in an oil property for the tested fuel contamination levels.

a. Viscosity

Table 17 lists possible allowable changes for viscosity [5]. Note that these limits placed on the change in viscosity are not specified for an engine or oil type. For the case in which the change in viscosity exceeds the specified changes listed in Table 3.5, it is likely that the oil has lost its ability to perform its desired functions. As previously mentioned, other property measurements should be considered before coming to this conclusion. Obviously the oil viscosity will change with temperature, but what's of importance in this section is the fuel effect on viscosity *at a given temperature*, so the temperature dependence, while fully acknowledged is removed for these measurements.

Viscosity measurements were conducted with both the on-board oil-condition sensor and according to ASTM D 445. A trend in viscosity for fuel contaminated oil was established for both sets of measurements. A comparison was made between the change in viscosity between baseline measurements with unused oil and measurements with fuel-contaminated oil, using both measuring techniques. These comparisons accomplished the first objective. The amount of fuel contamination that resulted in the viscosity changing to its minimum limit was compared to respective values from Table 17. This accomplished the second objective.

| Property | Allowable Change | Allowable Change | Allowable Change |
|-------------------|---------------------|-------------------|---|
| Viscosity @ 100°C | 10-18 cSt | From +25% to -18% | ± viscosity grade from new oil 3 or 4 cSt from new oil |

Table 17: Limits on the change in viscosity at 100°C [5]

Figure 23 shows viscosity measurements obtained with both the on-board oil-condition sensor and from ASTM D 445 at 100°C. Oil samples were contaminated with at least 0.5% fuel by volume and at most 10.5% fuel by volume. In both cases the viscosity shows a decrease, as expected.

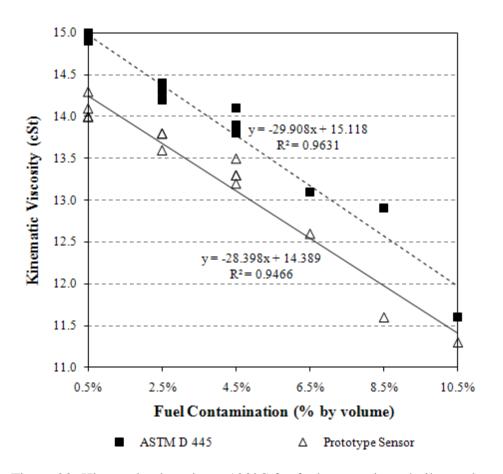


Figure 23: Kinematic viscosity at 100°C for fuel-contaminated oil samples

The coefficient of multiple determination R², is 0.96 for measurements conducted by ASTM D 445 and 0.95 for measurements conducted by the oil sensor. R² measures the reduction in the total variation between the observed viscosity and the viscosity associated with the regression model for the tested levels of fuel contamination. A high R² value in addition to a small precision error, as stated in Section 2.5b of Chapter 2, indicate that the proposed regression models shown in Figure 23 will be useful in quantifying the change in viscosity as a result of fuel contamination. It is important to note that here fuel was manually added to the oil, thereby eliminating other sources of contamination or oil degradation. In an engine operating environment there will never be a single source for a change in the physical properties of the engine oil. Any model illustrating changes in the physical properties of engine oil needs to consider all sources of oil degradation. The purpose in showing the regression models and the respective coefficient of multiple determination is to conclude if there is a significant difference between the output of the oil-condition sensor and the results from the accepted standardized testing method for viscosity. A significant difference in the models would suggest either unusually high experimental error or possible problems with the oil-condition sensor when it operates in a fuel contaminated environment. Either case would require further experiments to determine possible causes for the significant difference. As evidenced in Figure 23, the results from the ASTM standard and sensor measurements agree within 10%. This 10% is still less than the change in viscosity that is considered to be a condemning limit, +25% or -18% from the baseline value. Table 18 shows the viscosity measurements obtained from ASTM D 445.

| Kinematic Viscosity at 100°C (ASTM D 445) | | | |
|---|---|--------------------------|--------------------------------|
| Fuel Contamination (% by volume) | Viscosity for fuel-contaminated samples (cSt) | Baseline Viscosity (cSt) | Percent Change in Viscosity |
| 0.5 | 14.9 | 15.2 | 2.0 |
| 2.5 | 14.3 | 15.2 | 5.9 |
| 4.5 | 13.9 | 15.2 | 8.6 |
| 6.5 | 13.1 | 15.2 | 13.8 |
| 8.5 | 12.9 | 15.2 | 15.1 |
| 10.5 | 11.6 | 15.2 | 23.7 |

Table 18: Change in viscosity due to fuel contamination (ASTM D 445)

The first viscosity reading that indicates a condemning limit occurred at a fuel contamination level 10.5% by volume. This contamination is considered excessive (see Table 16). Figure 24 shows the viscosity of the fuel contaminated samples in comparison to the baseline measurements according to ASTM D 445. Table 19 shows the viscosity values obtained from the oil sensor.

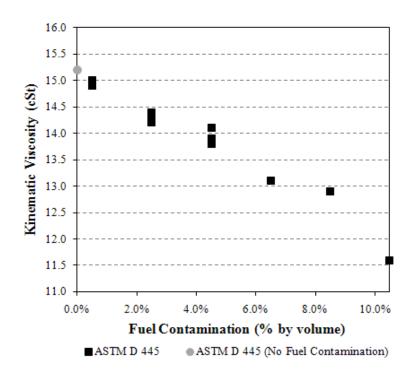


Figure 24: Comparison between baseline measurements and single-contaminated measurements (ASTM D 445)

| Kinematic Viscosity at 100°C (oil-condition sensor) | | | |
|---|---|--------------------------|--------------------------------|
| Fuel Contamination (% by volume) | Viscosity for fuel-contaminated samples (cSt) | Baseline Viscosity (cSt) | Percent Change in Viscosity |
| 0.5 | 14.1 | 14.4 | 2.1 |
| 2.5 | 13.7 | 14.4 | 4.9 |
| 4.5 | 13.3 | 14.4 | 7.6 |
| 6.5 | 12.6 | 14.4 | 12.5 |
| 8.5 | 11.6 | 14.4 | 19.4 |
| 10.5 | 11.3 | 14.4 | 21.5 |

Table 19: Change in viscosity due to fuel contamination (oil-condition sensor)

The first viscosity reading by the oil-condition sensor that indicates a condemning limit occurred at a fuel contamination level 8.5% by volume. This contamination is considered excessive by Table 16. Figure 25 shows the viscosity of the fuel

contaminated samples in comparison to the baseline measurements according to oil-condition sensor. Table 20 compares the viscosity measurements between the oil-condition sensor and ASTM D 445.

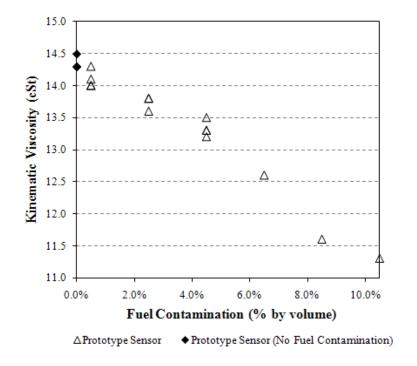


Figure 25: Comparison between baseline measurements and single-contaminated measurements (oil-condition sensor)

Table 20 shows that the results from ASTM D 445 were consistently higher than measurements from the oil-condition sensor, but the difference between the methods of measuring the viscosity are small. The greatest difference between the sets of measurements occurred with an oil sample that was contaminated with fuel by 8.5% by volume. At this contamination level the viscosity reading from ASTM D 445 was 11% greater than the viscosity measurement from the oil-condition sensor. Results from experiments with fuel-contaminated oil samples indicate that the oil-condition sensor is able to operate in a fuel contaminated environment.

| Kinematic Viscosity at 100°C | | | |
|----------------------------------|---|------------------|---|
| Fuel contamination (% by volume) | On-board oil- condition sensor (cSt) | ASTM D 445 (cSt) | Percent Difference between sensor and ASTM measurements (cSt) |
| 0.5 | 14.1 | 14.9 | 5.4 |
| 2.5 | 13.7 | 14.3 | 4.2 |
| 4.5 | 13.3 | 13.9 | 4.3 |
| 6.5 | 12.6 | 13.1 | 3.8 |
| 8.5 | 11.6 | 12.9 | 10.1 |
| 10.5 | 11.3 | 11.6 | 2.3 |

Table 20: Comparison between methods used to measure viscosity

Possible sources of error in viscosity measurements between the ASTM method and the sensor could be attributed to slight temperature differences (i.e., approximately two degrees) when conducting the measurements. To quantify this effect, ASTM D 341 was used to calculate the change in viscosity corresponding to a \pm 2 degree temperature difference. Results show that a difference in two degrees yields approximately a 5% change in the viscosity. Therefore in general, temperature differences account for the discrepancy between sensor and ASTM output.

b. TBN

The following results address the second objective, which is to verify correlations between contamination levels and changes in lubricant properties with respect to TBN. Table 21 lists the recommended limits and allowable change in TBN [5].

| Property | Recommended Limits | Allowable Change |
|----------|--------------------|------------------|
| TBN | 0.5-4 mg KOH/g | -50% |

Table 21: Limits on the change in TBN [5]

As seen in Table 21, the recommended limit placed on TBN is 0.5-4 mg KOH/g. This implies that if the measured TBN of a used oil sample is 0.5-4 mg KOH/g, then the oil is most likely no longer able to perform its desired functions. This TBN measurement should be considered in respect to other property measurements and parameters to assess the condition of the oil. Table 21 states the allowable change in TBN to be -50%. This implies that the TBN is allowed to decrease by 50% from the baseline measurement. A decrease greater than 50% indicates that the oil is likely to have lost its capability to perform as desired.

The change in TBN between baseline measurements using ASTM D 4739 and measurements with fuel-contaminated samples using ASTM D 4739 was compared to the specified, recommended limits and allowable changes shown in Table 21. Fuel was added to the oil samples in a concentration of 6% by volume, which is considered significant according to Table 16. The presence of fuel in oil will only lead to reactions between the additives in the oil and the fuel, eventually decreasing the TBN, if the oil is at a certain temperature. The temperature at which the additives become active in neutralizing acidic components for the Rotella® T SAE 15W-40 engine oil is 100°C. At high temperatures, oxidation of the fuel leads to the formation of sulfur oxides, due to the sulfur present in the fuel. When the sulfur oxides react with water, sulfuric acid is produced. The additives in the oil neutralize the sulfuric acid, eventually leading to a drop in the TBN as the additives become depleted. Since

temperatures greater than or equal to 100°C are needed to initiate the oxidation, eventually leading to a reduction in the additives, the samples (containing 6% by volume fuel) in these experiments were heated to 120°C and kept at this temperature for approximately one hour. It took three hours for the sample to cool to ambient temperature. The sample was covered with parafilm to avoid contamination and stored for TBN testing on the following day. Figure 26 shows the titration curves for fuel-contaminated oil samples.

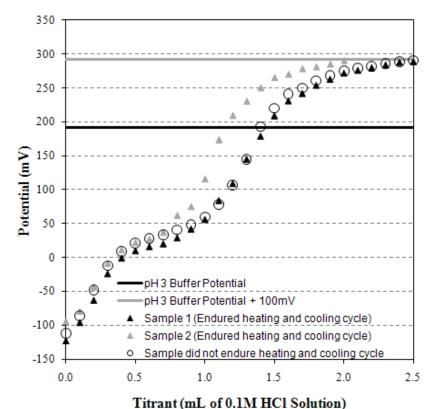


Figure 26: Titration curves for unused Shell oil with 6% fuel by volume

For each of the samples, the inflection point occurred at a potential less than the potential reading for the ph 3 buffer. This indicates that the acids in the oil were neutralized at a titrant volume less than what was needed to reach the potential of the

pH 3 buffer. The titration curves for each sample follow the same path for low and high potential readings. There is some deviation in the titration curves for potential readings in the approximate range of 50 - 275 mV. Table 22 shows the TBN values for the fuel-contaminated oil samples. The TBN values for the fuel-contaminated oil samples show a decrease from the baseline measurement, 12.4 ± 0.4 .

| | Sample 1 | Sample 2 | Sample 3 |
|-----|----------|----------|----------|
| TBN | 11.7 | 9.9 | 10.9 |

Table 22: TBN values for fuel-contaminated oil samples

Since TBN is affected by oxidation, tests were conducted with unused oil samples that were exposed to an environment that would induce oxidation. In this case, oxidation is considered as the source of oil degradation whereas previously fuel was the source of oil degradation by contamination. A beaker with approximately 50 mL of oil was placed in a convective furnace and continuously heated for fifteen days at approximately 120°C. After heating, the oil was allowed to cool to ambient temperature. Cooling was required because the ASTM standard specifies the testing at ambient temperature. Three oil samples were extracted from the beaker and the TBN was measured for each sample. The titration curves for these tests are shown in Figure 27.

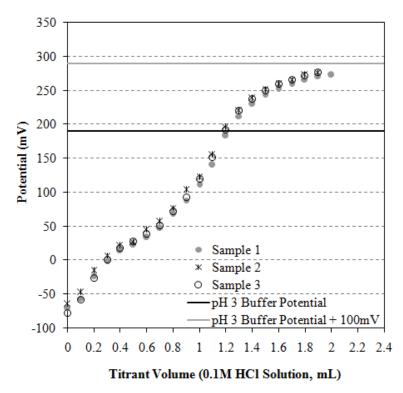


Figure 27: Titration curves for Shell engine oil heated for fifteen days

The titration curves follow the same path for each of the samples, with the inflection point occurring at a potential beneath the pH 3 buffer potential. The TBN values for these samples are shown in Table 23.

| TBN | | | |
|----------|------------------------|-----------------------------------|--------------------|
| | Oil heated for 15 days | Unused Oil (Baseline Measurement) | Percent Difference |
| Sample 1 | 11.0 | 12.4 | 12.7 |
| Sample 2 | 10.8 | 12.4 | 14.8 |
| Sample 3 | 10.7 | 12.4 | 15.9 |

Table 23: TBN for unused oil that was heated for fifteen days

The percent difference between the TBN values for the heated oil samples and the baseline measurements does not exceed the allowable change of -50% shown in Table

21. Also, the TBN of the oil that was heated did not decrease to a value within the range 0.5-4 mg KOH/g. Due to time and resource limitations, additional experiments involving longer durations and higher temperatures fall outside the scope of this investigation.

c. Dielectric Constant

No limits on the change in dielectric constant were found in the literature, although recommendations to evaluate the oil with conventional analysis when changes in dielectric constant exceed 0.01 were found [21] whereas other sources list changes in the dielectric constant of 0.2 as significant. So there is an order of magnitude difference between the values considered significant. Changes in dielectric constant from the baseline measurements using the on-board oil-condition sensor and the BI-870 bench-top instrument due to fuel contamination were compared to the specified, allowable changes shown in Table 5 to meet the first objective of this research. In addition, regression analysis was used to establish a trend in dielectric constant for fuel contaminated oil, meeting the second objective.

The BI-870 bench-top instrument measurements were compared to the measurements from the on-board oil-condition sensor using fuel-contaminated oil samples heated and stabilized at different temperatures. It has been suggested that fuel contamination should not result in drastic changes in the dielectric constant of the oil since the dielectric constant of both media are very similar [21]. The temperature to which the samples were heated was below 100°C, indicating that the additive activity was limited if there was any at all. If this were not the case, reactions between the additives in the fuel may result in changes in the dielectric constant.

Figures 28-29 show the dielectric constant as measured with both the BI-870 benchtop instrument and the oil-condition sensor for fuel-contaminated oil samples at various temperatures.

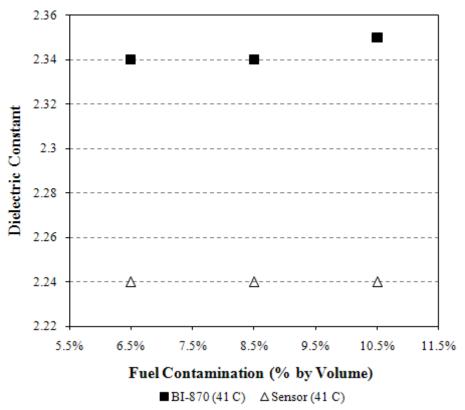


Figure 28: Dielectric constant of fuel-contaminated oil at 41°C

Figure 28 illustrates that the oil-condition sensor continues to show a bias in a fuel-contaminated environment at 41°C. The dielectric constant, as measured with the oil-condition sensor, shows no change as the amount of fuel contamination changes. The BI-870 bench-top instrument shows a 0.01 change when the fuel contamination increases from 8.5% to 10.5% by volume. Note that the BI-870 measures the dielectric constant with an accuracy of 0.01, so the observed change

shown in Figure 28 may be due to the limiting accuracy of the measurements from the BI-870.

Figure 29 compares the continuous change in the dieletric constant, as measured with the oil-condition sensor during a thermal cycle. Experimental set-up 1 was used, the oil sample was contaminated 6% by volume with diesel fuel. This experiment was conducted for two consecutive days, as shown in Figure 29, using the same beaker of oil sample. After the first day of testing, the beaker was stored until the second day. The purpose in conducting these experiments was to test for permanent changes in the dielectric constant for contaminated oil samples. Note that the rate of temperature increase and decrease was the same as for the previous experiments, (Figure 18) for the case of no fuel contamination.

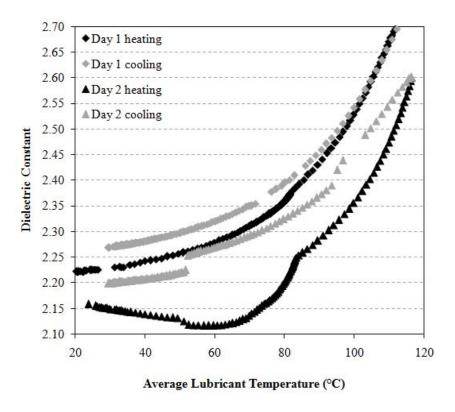


Figure 29: Change in dielectric constant for a fuel-contaminated oil sample (6% by volume) during a thermal cycle

The dielectric constant values corresponding to the first day of testing are the same for the heating and cooling phases at temperatures above 100°C. At temperatures less than 100°C, the dielectric constant values during the cooling phase are greater than those for the heating phase. The dielectric constant measurements during the second day were noticeably less than the measurements on the first day. During the heating phase for Day 2 the dielectric constant showed an initial decrease, prior to increasing, of approximately 0.05 from the starting measurement.

Figure 30 compares the measurements from the oil-condition sensor to measurements from the BI-870 bench-top instrument for fuel-contaminated (6% by volume) oil samples enduring a thermal cycle.

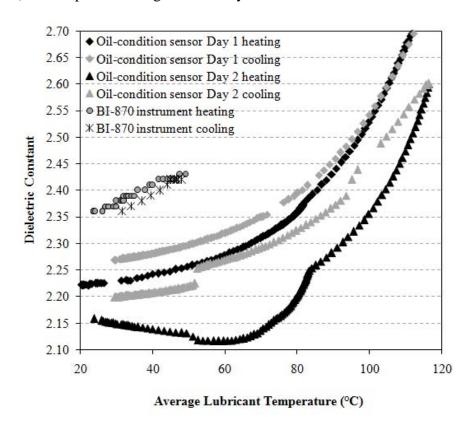


Figure 30: Change in dielectric constant for a fuel-contaminated oil sample (6% by volume) during a thermal cycle for BI-870 instrument and oil-condition sensor

Figure 30 shows the BI-870 instrument to exhibit much less change in the dielectric constant in comparison to measurements from the oil-condition sensor.

This may be attributed to the fact that the instruments operate at different frequencies, which has been cited as a possible cause for differences in measurements of the same fluid [20].

Another noticeable feature in Figure 30 is that dielectric constant measurements from the BI-870 bench-top instrument during the cooling phase of the thermal cycle are shifted beneath the measurements from the heating phase, the contrary is observed with the oil-condition sensor.

Figure 31 compares the dielectric constant measurements from the oil-condition sensor for the first day of inducing the oil sample to a thermal cycle for both non-contaminated and contaminated oil samples.

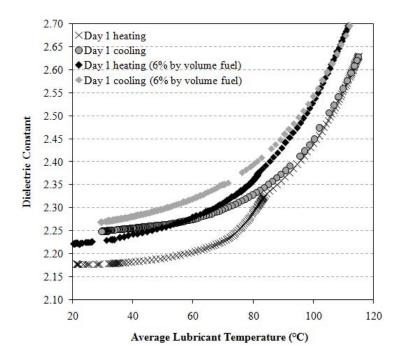


Figure 31: Change in dielectric constant for oil samples (non-contaminated and contaminated) during a thermal cycle as measured by the oil-condition sensor for Day 1 of testing

Dielectric constant measurements conducted with the fuel-contaminated oil sample are less than the measurements taken using the non-contaminated oil sample except for measurements during the cooling phase at approximately 55°C and less.

Figure 32 compares the dielectric constant measurements from the oil-condition sensor for the second day of inducing the oil sample to a thermal cycle for both non-contaminated and contaminated oil samples.

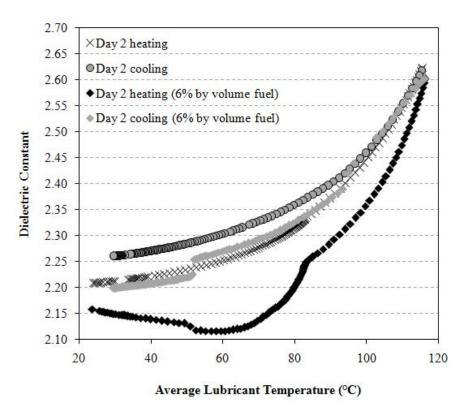


Figure 32: Change in dielectric constant for oil samples (non-contaminated and contaminated) during a second thermal cycle as measured by the oil-condition sensor for Day 2 of testing

The dielectric constant measurements during the second day of being induced to a thermal cycle show the same trend as observed on the first day, with the curve being shifted down. Since the rate of the temperature increase and decrease were nearly the same for both days it is most likely that the reason for the downward shift in the

dielectric constant is a result of permanent changes in the dielectric constant in addition to fuel contamination.

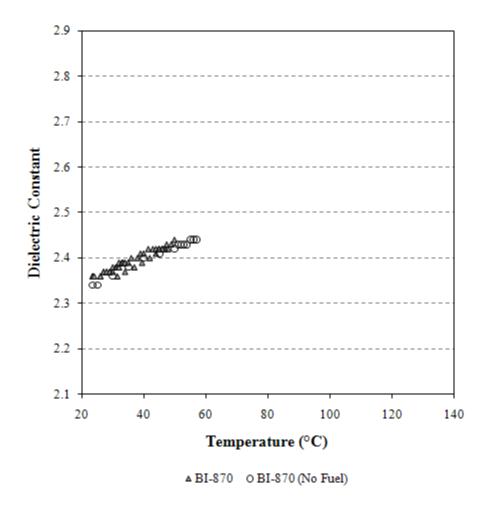


Figure 33: Change in dielectric constant for a fuel-contaminated oil sample (6% by volume) during a thermal cycle as measured by the BI-870 bench-top instrument

Figure 33 shows the dielectric constant as measured from the BI-870 bench-top instrument for unused oil and fuel-contaminated (6% by volume) oil samples during a thermal cycle as measured with the BI-870 bench-top instrument. Figure 33 shows that fuel contamination has a small, if any, effect on the dielectric constant based on the BI-870 bench-top instrument measurements.

d. Flash Point

The results from the flash point tests were used in order to estimate the amount of fuel contamination in oil samples from the engine. More accurate methods for detecting fuel contamination, such as Fourier Transform Infrared (FTIR) spectroscopy are available, but could not be used in this research due to cost limitations. Figure 34 shows that, as expected, the flash point decreases as fuel contamination increases. Note that the primary objective to conducting flash point measurements was for their use in accomplishing objective 4, see Section 1.2 of Chapter 1. Table 24 shows the flash point values for the fuel-contaminated oil samples.

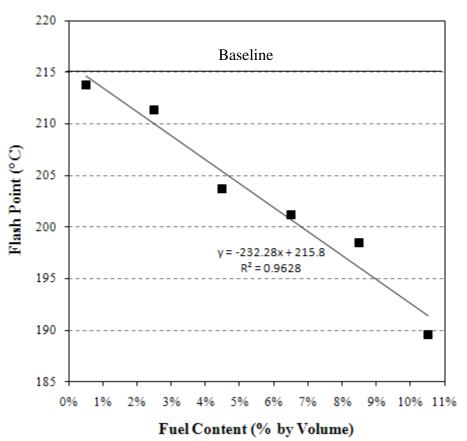


Figure 34: Flash point for fuel-contaminated oil samples

| Percent by volume of fuel contamination | Flash Point (°C) |
|---|------------------|
| 0.5% | 213.8 |
| 2.5% | 211.4 |
| 4.5% | 203.7 |
| 6.5% | 201.2 |
| 8.5% | 198.4 |
| 10.5% | 189.6 |

Table 24: Flash point values for fuel-contaminated oil samples

ENGINE EXPERIMENTS

4.1 Engine Experiments

The objectives of the engine experiments were to quantify how lubricant properties change with engine operating time, quantify correlations between measured properties and determine potential causes for any detected property changes. This chapter will proceed with introducing the engine set-up and describing the testing procedure, followed by a discussion on the findings.

4.2 Description of Engine Experiments

a. Engine Characteristics and Set-up

The engine used in this research was a 6.5-liter V-8, naturally aspirated diesel engine, shown in Figure 35. Maximum torque and power are listed as 290 ft-lbs @ 1800 rpm and 160/170 hp @ 3200 rpm [37].



Figure 35: 6.5 L V-8 naturally aspirated diesel engine

The engine operated using Shell Rotella T 15W-40 Advanced Soot Control diesel engine oil. It was equipped with an oil cooler and numerous sensors were installed to monitor engine operation. These included: thermocouples to measure exhaust gas temperatures; an intake air temperature sensor; and a throttle position sensor. The engine was coupled to a water-brake dynamometer (GoPower Systems' D557). The dynamometer was equipped with a load cell (Interface model SSM-1000) to measure brake torque. The load cell and sensors were connected to a data acquisition card (NI-SCXII). A virtual instrument (VI) was designed in Lab View, shown in Figure 36, to display the output from the sensors and load cell.

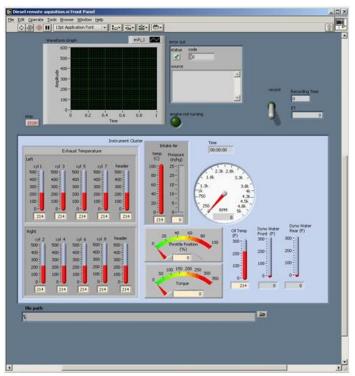


Figure 36: Lab View interface to monitor output from sensors and load cell

The on-board oil-condition sensor was mounted in the oil gallery prior to the oil filter. This maximized the contamination state of the oil prior to flowing through the

sensing element of the oil-condition sensor. Also, by being installed in the oil gallery, the sensor was operating in non-stagnant conditions.

The on-board oil-condition sensor communicated to a sensor control unit (SCU), which was connected to a lap top via an RS232 cable. Proprietary software recorded the sensor output to a text file and displayed the sensor output on the lap top.

b. Test Procedure

The engine warm-up period was approximately 20 minutes. This time was sufficient for the on-board sensor to reach thermal equilibrium. Once the coolant temperature reached 180°C, the engine speed and torque were increased to approximately 2200 RPM and 210 ft-lbs, respectively. The engine was operated for a total of 73 hours over the course of 12 days. The oil was sampled periodically in 150 mL amounts. A summary of tests is shown in Table 25.

| Day | Avg. Engine Speed (RPM) | Avg. Torque (ft-lbs.) | Sampling Time And Engine Operating Time |
|-----|---|---|--|
| 1 | 2209.88 ± 48.45 | 210.21 ± 2.63 | Sample 1: after warm-up Sample 2: end of the test Test Duration: 5 hrs Total Engine Run Time: 5 hrs |
| 2 | 2208.56 ± 66.96 | 206.39 ± 3.97 | Sample 1: after warm-up Sample 2: end of the test Test Duration: 7 hrs. Total Engine Run Time: 12 hrs. |
| 3 | 2225.56 ± 52.52 (during first 4 hrs.) 2263.90 ± 64.27 (during last 3 hrs.) | 210.40 ± 2.22 (during first 4 hrs.) 210.07 ± 1.94 (during last 3 hrs.) | Sample 1: after warm-up Sample 2: after 4 hrs. Sample 3: after 3 hrs. Test Duration: 7 hrs. Total Engine Run Time: 19 hrs. *added 2.4 quarts at end of day |

| 4 | 2219.60 ± 51.82 | 211.24 ± 2.56 | Sample 1: after warm-up Sample 2: end of the test Test Duration: 4 hrs. Total Engine Run Time: 23 hrs. |
|----|---------------------|-------------------|--|
| 5 | 2215.96 ± 57.95 | 213.43 ± 2.22 | Sample 1: after warm-up Sample 2: end of the test Test Duration: 8 hrs. Total Engine Run Time: 31 hrs. |
| 6 | 2173.69 ± 45.83 | 222.04 ± 1.60 | Sample 1: before warm-up Sample 2: end of the test Test Duration: 2 hrs. Total Engine Run Time: 33 hrs. |
| 7 | 2205.25 ± 44.87 | 208.73 ± 3.63 | Sample 1: before warm-up Sample 2: end of the test Test Duration: 8 hrs. Total Engine Run Time: 41 hrs. |
| 8 | 2234.98 ± 58.19 | 209.41 ± 2.24 | Sample 1: before warm-up *added 2.5 quarts after sample 1 Sample 2: end of the test Test Duration: 4 hrs. Total Engine Run Time: 45 hrs. |
| 9 | 2099.22 ± 51.01 | 217.88 ± 2.34 | Sample 1: before warm-up Sample 2: end of the test Test Duration: 8 hrs. Total Engine Run Time: 53 hrs. |
| 10 | 2167.16 ± 36.26 | 214.05 ± 2.58 | Sample 1: before warm-up Sample 2: end of the test Test Duration: 8 hrs. Total Engine Run Time: 61 hrs. |
| 11 | 2194.12 ± 45.34 | 211.24 ± 1.93 | Sample 1: before warm-up Sample 2: end of the test Test Duration: 5 hrs. Total Engine Run Time: 66 hrs. |
| 12 | 2313.35 ± 81.02 | 205.67 ± 2.69 | Sample 1: before warm-up *added 3.0 quarts after sample 1 Sample 2: end of the test Test Duration: 7 hrs. Total Engine Run Time: 73 hrs. |

Table 25: Summary of engine tests

4.3 Results

Figure 37 shows the dielectric constant as measured from the on-board oil-condition sensor over the course of engine testing.

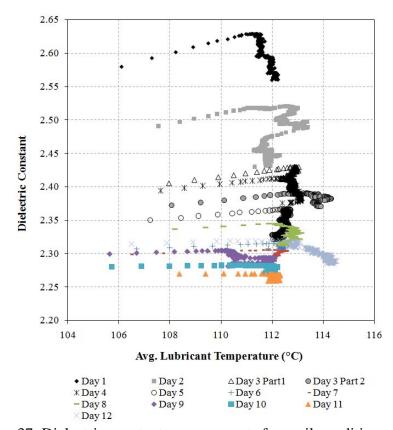


Figure 37: Dielectric constant measurements from oil-condition sensor

This figure also shows the trend in the dielectric constant within a given day and the change in the dielectric constant from one day to the next. Note that for a given day the dielectric constant initially increases as the lubricant temperature increases to its stabilization point. This initial increase could be attributed to the temperature increase and to additives within the oil becoming active. Note that this increase in dielectric constant for an increase in temperature was also observed with the bench-top experiments where the lubricant was subjected to a thermal cycle.

To process the data from the twelve days of testing, the lubricant temperature and dielectric constant values were averaged within the stabilization zone. Results are shown in Figure 38.

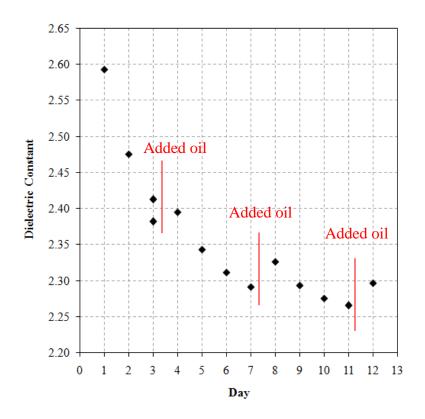


Figure 38: Dielectric constant as a function of engine test day

It can be seen that the change in the dielectric constant from day to day is greater than could be attributed to a temperature effect. The dielectric constant decreases as engine testing progressed. An increase in the dielectric constant for used oil was expected due to the accumulation of contaminants and acidic by-products from oxidation during oil degradation. To investigate the decrease in dielectric constant, soot measurements were conducted on samples gathered during the second and final days of testing. The soot concentration remained constant at 0.1%. The low soot content supports the observed

decrease in dielectric constant. Changes in oil density throughout the course of engine testing were also investigated. As mentioned in Chapter 1, a decrease in density would lead to a decrease in dielectric constant. As shown in Figure 39 however, the density increases, which does not support the observed trend.

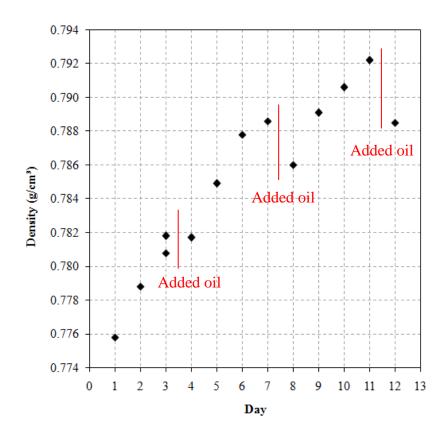


Figure 39: Density as a function of engine test day

This decrease in the dielectric constant might be attributed to incipient consumption of the additive package. As Figure 38 shows, the addition of new oil resulted in an increase in the dielectric constant, which validates this conclusion.

Figure 40 shows the kinematic viscosity as measured with the on-board oil-condition sensor for the duration of the engine tests. Similar trends for the kinematic viscosity were

observed for each subsequent day, showing an increase in the kinematic viscosity as the engine testing progressed.

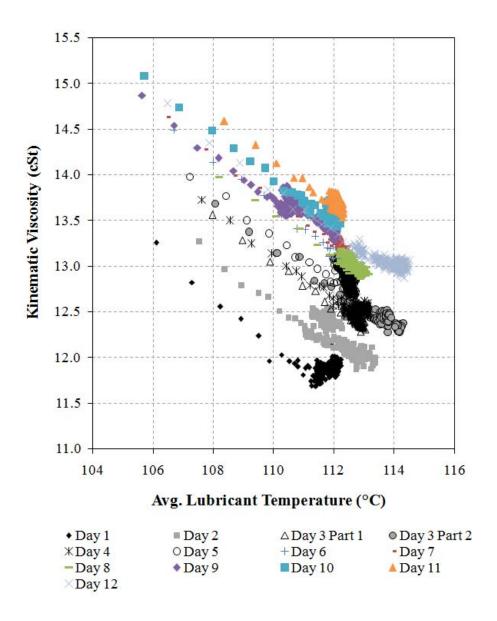


Figure 40: Kinematic viscosity measurements from oil-condition sensor

Figure 41 shows the trend in the oil kinematic viscosity over the entire testing period.

Note that for a given day the kinematic viscosity continuously decreases until the temperature stabilizes. As previously discussed (see Chapter 1), there are numerous

factors that cause a change in the kinematic viscosity, or no change if there are canceling effects such as both soot and fuel contamination. Viscosity does have a temperature dependence, but the change in kinematic viscosity due to a temperature change was found to be much less than the change in kinematic viscosity from a day to day basis, as was the case with the dielectric constant measurements. This allowed for the temperature dependence to be removed from this set of data and to show the kinematic viscosity as a function of time, shown in Figure 41.

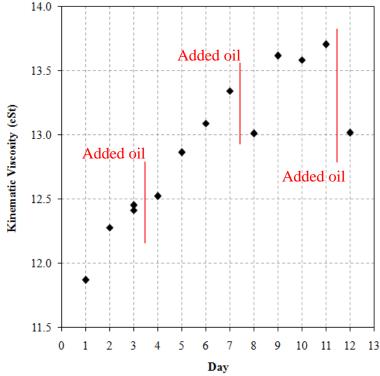


Figure 41: Kinematic viscosity as a function of engine test day

Figure 41 shows the kinematic viscosity to increase as engine testing progressed. The addition of new oil resulted in small but significant changes in the viscosity on Days 8 and 12. The increase in kinematic viscosity over the course of engine testing is a likely result of contamination from by oxidation by-products.

Figure 42 shows the dielectric constant plotted as a function of kinematic viscosity. Both lubricant property values were obtained from the oil-condition sensor over the course of the engine testing and were averaged over the stabilized temperature zone. This figure shows that there is a correlation between dielectric constant and viscosity changes.

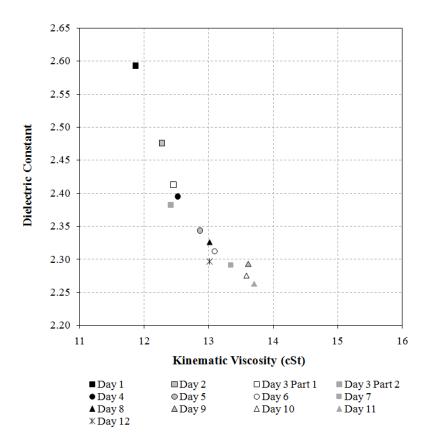


Figure 42: Dielectric constant shown as a function of kinematic viscosity

The observed correlation between the dielectric constant and the viscosity could possibly be explained by the depletion of the additives having a dominating effect over the increase in the oxidation by-products. Depletion of the additives would explain a decrease in the dielectric constant. Increase in oxidation by-products would explain the increase in the viscosity.

In order to investigate possible causes for the observed changes in the dielectric constant and kinematic viscosity measurements TBN and flash point measurements were conducted. Table 26 shows the change in TBN from beginning to end of engine testing.

| Engine Baseline | Day 1 | Day 12 |
|-----------------|-------|--------|
| 12.9 ± 0.6 | 11.0 | 10.7 |

Table 26: Summary of TBN measurements from engine experimentation

The engine baseline TBN is the average TBN of three oil samples that were collected after the warm-up period. The TBN shown for Day 1 was measured from an oil sample taken at the end of the five hours of engine operation on Day 1. The TBN shown for Day 12 was measured from an oil sample taken at the end of the seven hours of engine operation on Day 12. At this point, the engine had accumulated 73 hours of runtime. The TBN of Day 1 decreased by 14.7% from the engine baseline TBN. This is less than the decrease of 50%, which was previously referenced in Chapter 1 as a condemning limit for TBN. The TBN of Day 12 decreased by 17.1% from the engine baseline TBN and only 2.7% from Day 1. The small decrease in the TBN between the first and last day of testing is a likely result of the new oil that was added to the engine immediately before the final hours of engine testing on Day 12.

A decrease in the TBN over the course of engine testing would be expected due to the consumption of the oil additives, which would tend to decrease the dielectric constant and accumulation of acidic oxidation by-products, which would increase the lubricant viscosity.

An estimate of fuel contamination was obtained by conducting flash point measurements from samples collected during Days 11 and 12 of testing, as shown in

Table 27. A significant level of fuel contamination would decrease the lubricant flash point.

| Engine Baseline | Day 11 | Day 12 |
|-----------------------------|--------|--------|
| $217 \pm 1^{\circ}\text{C}$ | 225°C | 227°C |

Table 27: Summary of flash point measurements from engine experimentation

The engine baseline flash point is the average flash point of three oil samples that were collected after the warm-up period. The flash point shown for Day 11 was measured from an oil sample taken at the end of the five hours of engine operation on Day 11. At this point the engine had accumulated 66 hours of runtime. The flash point shown for Day 12 was conducted with an oil sample taken at the end of the seven hours of engine operation on Day 12.

The flash point of Day 11 increased by 3.4% from the engine baseline flash point. The flash point of Day 12 increased 4.6% from the engine baseline measurement. The trend in the flash point certainly does not indicated fuel contamination. The increase may be attributed to evaporation of lighter weight molecules during engine operation, leaving behind heavier weight molecules in the oil. Heavier weight molecules would require a greater temperature to evaporate, resulting in a higher flash point. However, the variation is within the uncertainty of flash point measurements, which precludes reaching a definite conclusion.

It needs to be noted that during bench-top measurements of flash point with manually contaminated oil samples the smallest contamination that was investigated was 1%. Flash point tests require 75 mL of oil, 1% of 75 mL (i.e., 0.75 mL) is a very small amount in comparison to the required amount of oil, approximately 11 quarts, to properly operate this engine. 1% of fuel contamination by volume of 11 quarts is 104 mL out of

10,400mL, this amount of fuel contamination would most likely go undetected in a flash point test.

In summary, the dielectric constant was found to decrease and the kinematic viscosity showed an increase over the course of engine operation. Fuel contamination is not likely to account for the observed trends. The initial decrease in the TBN supports the observed decrease in the dielectric constant, based on incipient depletion of the additive package. The increase in the kinematic viscosity may be a result of the accumulation of the oxidation by-products. No soot contamination was measured over the engine operating time.

CHAPTER 5

SUMMARY, CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE WORK

5.1 Summary and Conclusions

The goal of this research project was to quantify the condition of engine oil through direct, real-time measurements of the lubricant physical properties in an engine using an on-board oil-condition sensor. The objectives and the respective results of the project were:

- Validate the accuracy and precision of the lubricant properties measured by the on-board oil-condition sensor.
 - a. The temperature measurement from the on-board oil-condition sensor was within 1 to 2 degrees of a thermometer reading once the sensor reached thermal equilibrium, which takes approximately twenty minutes, and for temperatures above 55°C. The viscosity measurements from the oil-condition sensor were 5.3% less than the ASTM measurements at 100°C. The dielectric constant measurements from oil-condition sensor were 6.5% less than the bench-top instrument at stabilized temperatures.
- 2. Establish correlations between contamination levels and changes in lubricant properties.
 - a. The viscosity measurements from the oil-condition sensor and the ASTM method decreased as the fuel contamination increased, showing an agreement of 10 % between the sensor and the ASTM method

- 3. Quantify how lubricant properties change with engine operating time.
 - a. The dielectric constant decreased over engine operating time and increased upon the addition of new oil. The kinematic viscosity increased over engine operating time and decreased upon the addition of new oil. The decrease in the TBN, implying additive depletion, and the low soot content (0.1%) from the last oil sample are likely explanations for the decrease in the dielectric constant. In addition, no fuel contamination was detected, supporting the increase in the viscosity.
- 4. Determine correlations between measured properties and potential causes for any detected property changes.
 - a. A correlation was observed between the dielectric constant and the kinematic viscosity, further testing is needed to confirm if the observed correlation remains as oil degradation progresses. An initial decrease in the TBN supports the decrease in the dielectric constant due to depletion of additives and an increase in viscosity due to accumulation of oxidation by-products. Flash point measurements did not indicate fuel dilution.

5.2 Recommendations for Future Work

It was discussed in Chapter 1 that many oil-condition sensors use the dielectric constant to quantify the degradation of the oil, which emphasizes the usefulness of this property. Whereas there are published thresholds on the viscosity and TBN, the only

found thresholds on the dielectric constant are within disagreement and were certainly and quickly surpassed as this worked demonstrated. A significant contribution that could be made to the field of oil analysis would be the establishment of a threshold or a limit in the change for the dielectric constant. This research showed that the dielectric constant has a dependence on the engine oil and the sensor, but the work does not allow for clear, concise conclusions to be drawn on the threshold of the dielectric constant.

Future work could also include quantifying correlations between the thresholds of the dielectric constant and other lubricant properties. It is often the case that multiple properties are evaluated in oil analysis since each property of the lubricant reveals unique information concerning the capability of the lubricant to perform its required function.

Correlations between the thresholds would reveal what to expect in terms of oil degradation. For the case in which only a single lubricant property is able to be measured, due to cost constraints, these correlations would be extremely useful. Using a statistical technique such as response surface methodology would be useful to establishing such correlations, not only between lubricant properties but also between concentration of contaminants, and operating conditions. This statistical technique would be useful in analyzing the data to show how the lubricant properties (i.e., response variables) change with a specific change in levels of contamination or changes in operating conditions. In addition, this technique can be used to evaluate which factors (e.g., fuel dilution or soot contamination) have a dominating effect or if at a certain level of contamination there is an interaction between the factors resulting in a canceling effect.

The challenge in implementing this technique is keeping all other factors not being tested constant. Response surface methodology, although certainly not necessary, could

be used to evaluate the change in lubricant properties for multiple oil types (e.g., gasoline oils, transmission oils, etc.). Tests with various oil types would also provide a more comprehensive understanding of the sensor capability.

In addition to looking at correlations between thresholds, correlations between trends in the lubricant properties, such as the dielectric constant and kinematic viscosity, could be quantified over longer test periods. This would reveal if the observed correlation between dielectric constant and kinematic viscosity stays the same as oil degradation progresses through continual engine operation. FTIR measurements with the collected oil samples would be useful in explaining the correlation between the lubricant properties.

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